

# **Bereskin & Parr**

INTELLECTUAL PROPERTY LAW

Appl. No : 09/621,234 Confirmation No.: 3325  
Applicant : HENSHAW et al.  
Filed : July 20, 2000  
Title : VERTICAL CYLINDRICAL SKEIN OF HOLLOW FIBER  
MEMBRANES AND METHOD OF MAINTAINING CLEAN FIBER  
SURFACES  
TC./A.U. : 1797  
Examiner : FORTUNA, Ana M.  
Docket No. : 4320-241  
Customer No. : 001059

Board of Patent Appeals and Interferences  
United States Patent and Trademark Office  
P. O. Box 1450  
Alexandria, Virginia 22313-1450

May 30, 2008

## **BRIEF IN SUPPORT OF APPEAL**

### **Real Party in Interest**

The Real Party in Interest is Zenon Technology Partnership, the assignee of the patent under appeal. Zenon Technology Partnership is a partnership of GE Betzdearborn Canada Company and 1244734 Alberta ULC. GE Betzdearborn Canada Company and 1244734 Alberta ULC are related to GE Betz Inc., MRA Investments Inc., MRA Systems Inc., GE Investments Inc. and General Electric Company.

### **Related Appeals and Interferences**

Appeals were filed in Application Serial Nos. 11/008,977, 11/049,988 and 11/059,403.

Manulife Corporate Park, 6733 Mississauga Road, Suite 600, Mississauga, Ontario L5N 6J5  
Tel: 905.812.3600 Fax: 905.814.0031 [www.bereskinparr.com](http://www.bereskinparr.com)

**T O R O N T O M I S S I S S A U G A W A T E R L O O M O N T R E A L**

These applications claim priority from Application Serial No. 08/690,045, which issued as Patent No. 5,783,083. The present application seeks to reissue Patent No. 5,783,083. These appeals were concluded either by way of the Examiner re-opening prosecution or by way of the Applicants filing an RCE without there being any decisions on the Appeals. A previous appeal in this application was terminated by way of the Applicants filing a RCE before the appeal was docketed.

### **Status of Claims**

Claims 1-18 and 24 are pending. Claims 19-23 have been cancelled. Claims 1-14 are allowed. Claims 15-18 and 24 are rejected and the subject of this appeal. A copy of the appealed claims appears in the Claims Appendix.

No amendments have been filed after the rejection of November 1, 2007.

### **Summary of the Claimed Subject Matter**

All references in the following paragraphs apply to either the application as filed or to Patent No. 5,783,083, a copy of which is attached in the Evidence Appendix.

The appealed claims relate to a hollow fiber membrane filtration system used to filter or separate a permeate from a liquid substrate having particulate matter therein (column 5, line 58 to column 6, line 6).

The system has a non-pressurized reservoir (also called tank 90, column 17, lines 24 and 35; Figures 11 and 12) for containing the substrate. An assembly having a plurality of hollow fiber membranes is immersed in the substrate. Examples of such assemblies are shown in Figures 2, 3 and 6-9. One example of an assembly is skein 80, having fibers 12 and upper and lower end caps 81, 82 (see also Fig. 8, and col. 15, lines 40-46). Another example of a membrane assembly is skein 10, with upper and lower end caps 21, 22, hollow fiber membranes 12 (which may be called "membranes" or "fibers"),

and upper and lower solid bodies 23, 24 (called headers, see column 22, lines 36) with corresponding ends of the fibers fixed therein (see Fig. 2, and col. 11, lines 25-59).

The membranes are sealingly secured in the solid bodies so as to prevent solids in the substrate from contaminating the permeate (column 7, lines 35-40). The solid bodies are comprised of a potting material, and at least a portion of the membranes are spaced apart by the potting material to a center to center distance in the range of 1.2 to 5 times the outside diameter of the membranes (column 7, lines 28-31). With such a spacing, the membranes do not touch each other but rather are spaced apart by the potting material between them (Column 11, lines 43-47).

The system includes a "permeate pan" (column 4, line 65) in fluid communication with the lumens of the hollow fiber hollow fiber membranes. The permeate pan comprises, in one example, structure defining an upper permeate collection zone 28 provided by way of an upper end cap 21 (Fig. 2; col. 11, lines 26-29; col. 12, lines 41-44). A permeate pan can also define a lower permeate collection zone 29 in the lower end cap 22 (column 11, line 32). In other examples the permeate pan comprises a stainless steel permeate pan 61 (Fig. 6; col. 14, lines 50-52).

The membranes have a defined length which is in the range of 0.1% to 5% greater than the distance between opposed surfaces of the solid bodies (column 8, lines 64-65).

A pump (column 17, lines 62-66) applies a suction to the lumens of the hollow fiber membranes to draw a component of the substrate (the permeate) through the membranes while leaving particulate matter in the substrate (column 17, lines 51-54).

A gas distribution system has through passages with openings distributed both radially and circumferentially between the membranes to discharge gas into the substrate and provide bubbles in the substrate. In one example, an air feed tube 44 communicates with arms 41 having discharge openings 43 therealong (Fig. 3; Fig. 3A; col. 12 lines 5-

9). In another example, air supply tube 86 has ports 104 that may be threaded to receive the arms of a spargers (Fig. 8; col. 15 line 40-col. 16 line 36).

### **Grounds of Rejection to be Reviewed on Appeal**

This is a broadening reissue application filed within two years of the issue date of the patent being reissued. Claims 15-18 and 24 were rejected under 35 USC 251 for being improperly broadened. The rejection in the Office Action dated November 1, 2007 incorporates paragraph 6 of the rejection of March 4, 2003 and the Examiner's Answer of December 1, 2006.

The essence of the rejection is an allegation of improper recapture. In particular, the rejection involves claims 1 and 9 in 08/690,045, now patent 5,783,083, which have the following provision:

each said header having said fibers spaced apart by a flexible support means having a thickness corresponding to a desired lateral spacing between adjacent fibers, said support means extending over only each terminal portion of said fibers near their ends, so as to maintain said ends in closely-spaced apart relationship.

This provision was added in an amendment filed January 12, 1998, a copy of which is attached in the Evidence Appendix. This amendment effectively cancelled prior versions of claims 1 and 9 that did not have the provision described above. The amended claims 1 and 9 were allowed and issued in patent 5,783,083. The Examiner argues that, since claims 15-18 and 24 in this reissue application lack the provision copied above, that these claims should be rejected under 35 USC 251 as involving an improper attempt to recapture surrendered subject matter.

### **Argument**

The Applicants submit that there is no improper recapture. Recapture is measured against the claims effectively cancelled by the January 12, 1998 amendment. Claims 15-18 and 23 are narrower than the claims prior to the January 12, 1998 amendment because they have at least a broadened form of the limitation added by that amendment. The present claims accordingly fit within MPEP 1412.02 I(c) 2(d) and do not improperly recapture surrendered subject matter.

The Applicants will repeat the relevant limitations of the cancelled claims, the claims of Patent No. 5,783,083 and claims 15-18 and 24 of the present application below:

Cancelled Claims (claims prior to the amendments of January 12, 1998):

No limitations regarding fiber spacing.

Claims 1 and 9 of Patent No. 5,783,083 with limitation added on January 12, 1998:

each said header having said fibers spaced apart by a flexible support means having a thickness corresponding to a desired lateral spacing between adjacent fibers, said support means extending over only each terminal portion of said fibers near their ends, so as to maintain said ends in closely-spaced apart relationship

Present Application Claims 15-18 and 24:

at least a portion of the membranes spaced apart from adjacent membranes by the potting material to a center to center distance in

the range from 1.2 to 5 times the outside diameter of the membranes

MPEP 1412.02 I refers to a three step test for recapture. While the Applicants disagree with at least some of the statements regarding the third step in the MPEP, for the purpose of this appeal, the Applicants will structure their arguments to follow the three step test.

The first step in the three step test for recapture is to determine in what respect the claims of the patent being reissued differ from the claims of the reissue application. The Examiner notes that the words "flexible support means" occur in the claims of the issued patent but not claims 15-18 and 24 of this application. The Examiner argues that the words "flexible support means" in the claims of Patent No. 5,783,083 are limited to "flexible planar support means such as strips or cards..." The Applicants disagree. The words "planar...such as strips or cards" are not in the claims of Patent No. 5,783,083. While strips or cards are mentioned as examples of flexible support means in the application, there is no basis for reading these words from the specification into the claims. However, regardless of the construction of "flexible support means", the first step alone is not determinative of recapture. This application was filed within two years of the issue of Patent No. 5,783,083 and so broadening is permissible. A broadening aspect may exist without recapture. Instead, any broadening aspect identified in the first step must be considered further under the second and third tests.

The second step involves determining if there was a surrender of subject matter and whether there is any broadening of the reissue claims in the area of the surrendered subject matter. A claim having no limitations at all as to fiber spacing was surrendered in application no. 08/690,045. Claims 15-18 and 24 of this reissue application retain a limitation from claims 1 and 9 of patent 5,783,083

relating to fiber spacing, in particular that the fibers are closely spaced apart (and within a numerical range of spacing), but without a reference to "flexible support means". In summary, there was a surrender of subject matter in the area of fiber spacing and omitting of the "flexible support means" from present claims 15-18 and 24 is a broadening aspect in the subject area of fiber spacing.

The third step asks whether the reissue claims are materially narrowed compared to the cancelled claims so as to avoid the recapture rule. The Applicants submit that the claims of the present application are not as broad or broader than the canceled claims in the area of surrender. On the contrary, the claims of the present application are narrower than the claims of U.S. Patent No. 5,783,083 in that they specify that the membranes are closely spaced apart and give a numerical range for the spacing of the membranes. Accordingly, the Applicants submit that the claims of this reissue application do not constitute recapture. Claims 15-18 and 24 at least contain a broadened form of the limitation added to secure allowance of patent No. 5,783,083. Thus, claims 15-18 and 24 present no recapture pursuant to MPEP 1412.02 IC2(d) and *Ex Parte Eggert*, 67 USPQ 2d 1716 (Bd.Pat.App.Intel.2003), see for example page 1717 left column and first three lines of the right column.

The Examiner states that the "flexible support means" is an omitted limitation. The Applicants respectfully submit that this comment is relevant to the second step, but does not correctly apply the third step. The third step requires comparing the reissue claims (present application claims 15-18 and 24) with the claims cancelled from the original application (08/690,045) by amendment to secure Patent No. 5,783,083. The cancelled claims did not have a "flexible support means". Accordingly, it is irrelevant in a discussion of the third step, that "flexible support means" has been omitted. Those words did not appear in the

claims of application No. 08/690,045 before the final amendment that resulted in patent No. 5,783,083 and so omitting those words does not recapture surrendered subject matter.

The Examiner further refers to the "flexible support means" as being an "essential element". The Applicants submit that a "flexible support means" is not an "essential element". For example, column 7, lines 35-42 of Patent No. 5,783,083 say that the choice of potting method is not critical. For further example, column 14, lines 28-31, describe a potting process in which flexible support means are used to space the membranes but are not part of the header. In any event, whether an element is "essential" is not part of the test for recapture.

The Examiner further states that the Applicants argued in an Amendment/Response filed on 1/12/1998 that membranes spaced by a flexible support means provided an advantage over prior art with fibers spaced by some other means. The Applicants respectfully disagree. Referring to pages 10 to 12 of that response, the Applicants argued that the prior art Kunio device had membranes that were in contact with each other and so not spaced at all. The present claims 15-18 and 23 are narrower than the claims prior to the Amendment/Response of 1/12/1998 in that they require the membranes to be spaced within a defined range of spacing. Accordingly, the present claims are consistent with the Applicants prior Amendment/Response.


### **Summary**

For the foregoing reasons, the Appellants believe that the Examiner's rejections of claims 15-18 and 24 are erroneous and respectfully request reversal of those rejections.



Respectfully submitted,

HENSHAW et al.

By 

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Scott Pundsack  
Reg. No. 47,330  
Tel: 416-957-1698

## CLAIMS APPENDIX

Claim 15. A system for withdrawing permeate from a liquid substrate while leaving particulate matter therein, comprising,

- (a) a non-pressurized reservoir other than a shell of a module for containing the substrate;
- (b) an assembly having a plurality of hollow fiber filtering membranes immersed in the substrate each membrane having a length greater than 0.5 m, the membranes together providing a surface area of at least greater than 1 m<sup>2</sup> and disposed generally vertically between upper and lower generally cylindrical solid bodies comprised of a potting material with (i) the solid bodies having the membranes sealingly secured therein so as to prevent the substrate from contaminating the permeate, at least a portion of the membranes spaced apart from adjacent membranes by the potting material to a center to center distance in the range from 1.2 to 5 times the outside diameter of the membranes, (ii) lumens of said membranes being in fluid communication with a permeate pan connected to one of the solid bodies and immersible in the substrate or to a pair of permeate pans connected one to each of the solid bodies and both immersible in the substrate, and, (iii) said membranes having a length between opposed surfaces of the solid bodies, in the range from 0.1% to 5% greater than the distance between opposed surfaces of the solid bodies;
- (c) a pump in fluid communication with said lumens of said membranes through at least one permeate pan, said pump operable to apply a suction to the lumens of the membranes to draw a component of the substrate as permeate through said membranes while leaving particulate matter in said substrate; and,
- (d) a gas-distribution system having through-passages with openings distributed both radially and circumferentially between the membranes

operable to provide a flow a gas through the through-passages to produce bubbles in the substrate.

Claim 16. The system of claim 15 wherein the length is in the range from 0.1% to 1% greater than the distance between the opposed surfaces of the [headers] solid bodies.

Clam 17. The system of claim 16 wherein the gas distribution system further includes a rigid air supply tube for carrying air to the through-passages and for spacing and positioning the lower and upper solid bodies relative to one another.

Claim 18. The system of claim 17 wherein the air supply tube has additional through-passages along its length.

Claim 24. The system of claim 15 wherein lower ends of the membranes are plugged.

## **EVIDENCE APPENDIX**

1. U.S. Patent No. 5,783,083
2. Amendment dated January 12, 1998 in Application Serial No. 08/690,045

**RELATED DECISIONS APPENDIX**

None



US005783083A

**United States Patent** [19]

Henshaw et al.

[11] Patent Number: 5,783,083

[45] Date of Patent: \*Jul. 21, 1998

[54] **VERTICAL CYLINDRICAL SKEIN OF HOLLOW FIBER MEMBRANES AND METHOD OF MAINTAINING CLEAN FIBER SURFACES**

5,403,479 4/1995 Smidt et al. 210/636  
5,480,553 1/1996 Yamamori et al. 210/650  
5,607,593 3/1997 Cote et al. 210/650  
5,639,373 6/1997 Mahendran et al. 210/636

[75] Inventors: Wayne Jerald Henshaw, Burlington;  
Mallivaganam Mahendran, Hamilton;  
Henry Behrmann, Puslinch, all of  
Canada

Primary Examiner—Ana Fortuna  
Attorney, Agent, or Firm—Alfred D. Lobo

[73] Assignee: Zenon Environmental Inc., Ontario,  
Canada

[\*] Notice: The term of this patent shall not extend  
beyond the expiration date of Pat. No.  
5,639,393.

[21] Appl. No.: 690,045

[22] Filed: Jul. 31, 1996

**Related U.S. Application Data**

[63] Continuation-in-part of Ser. No. 514,119, Aug. 11, 1995,  
Pat. No. 5,639,373.

[51] Int. Cl.<sup>6</sup> B01D 61/00

[52] U.S. Cl. 210/636; 210/500.23; 210/650;  
210/321.69; 210/356; 210/321.8; 210/321.89;  
210/257.2

[58] Field of Search 210/636, 321.69,  
210/321.8, 321.89, 257.2, 650, 500.23,  
641, 356

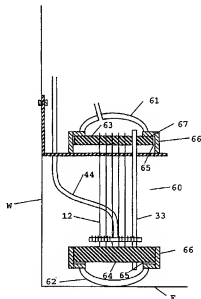
**References Cited****U.S. PATENT DOCUMENTS**

5,248,424 9/1993 Cote et al. 210/636

**ABSTRACT**

A gas-scrubbed vertical cylindrical skein of "fibers" has their opposed terminal portions held in headers unconfined in a modular shell, and aerated with a cleansing gas supplied by a gas-distribution means which produces a mass of bubbles serving the function of a scrub-brush for the outer surfaces of the fibers. The skein is surprisingly effective with relatively little cleansing gas, the specific flux through the membranes reaching an essentially constant relatively high value because the vertical deployment of fibers allows bubbles to rise upwards along the outer surfaces of the fibers. The effectiveness is critically dependent upon the length of each fiber in the skein. That length is in the range from at least 0.1% more than the fixed distance between opposed faces of the skein's headers, but less than 5% greater than the fixed distance. Lack of tension allows the fibers to sway in bubbles flowing along their outer surfaces making them surprisingly resistant to being fouled by build-up of deposits of inanimate particles or microorganisms in the substrate. For use in a large reservoir, a bank of skeins is used with a gas distributor means which has fibers preferably >0.5 meter long, which together provide a surface area >10 m<sup>2</sup>. The terminal end portions of fibers in each header are kept free from fiber-to-fiber contact with a novel method of forming a header.

14 Claims, 13 Drawing Sheets



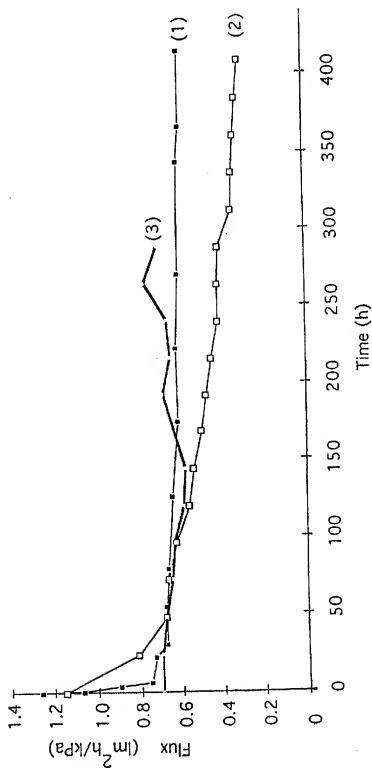
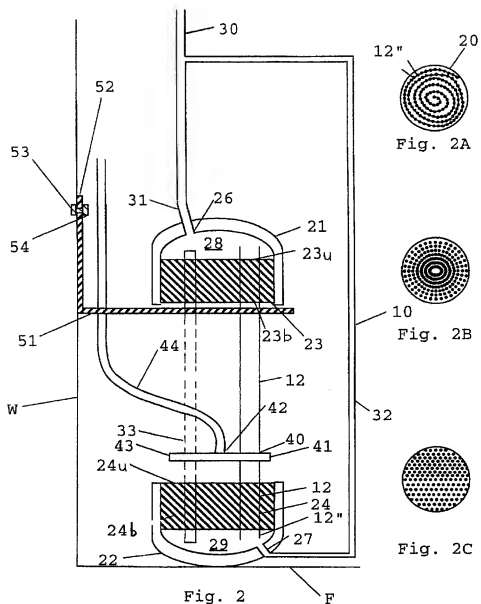


Figure 1





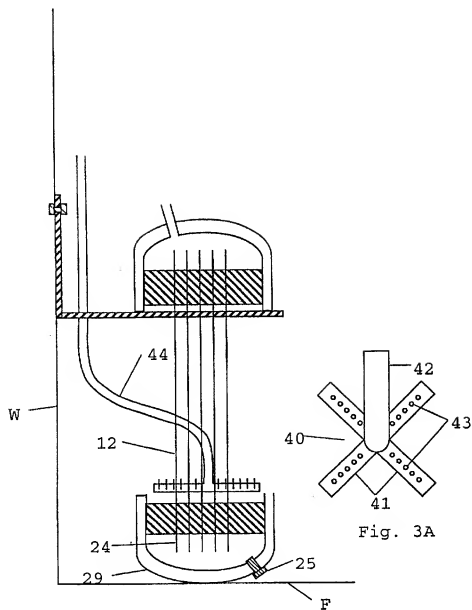


Fig. 3

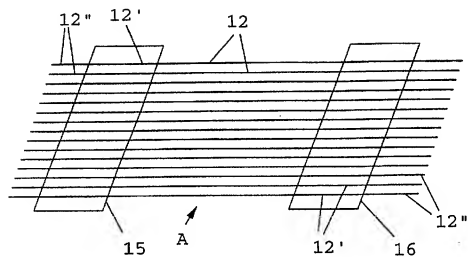


Fig. 4

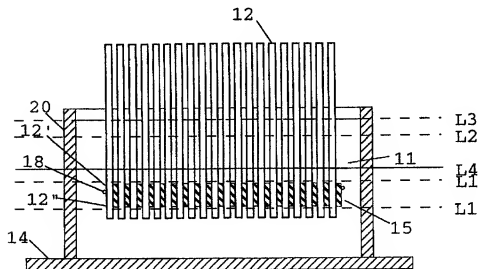


Fig. 5

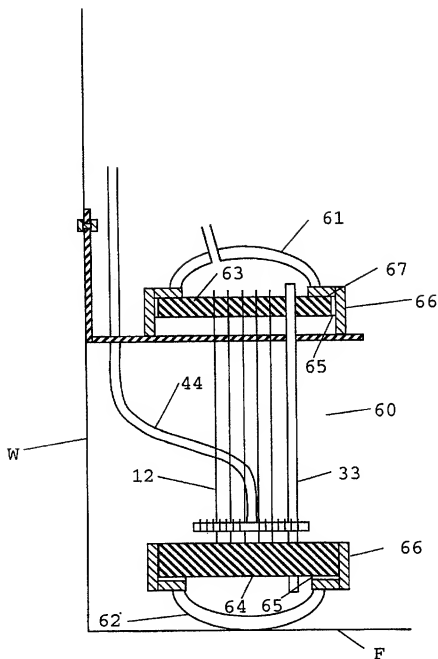


Fig. 6

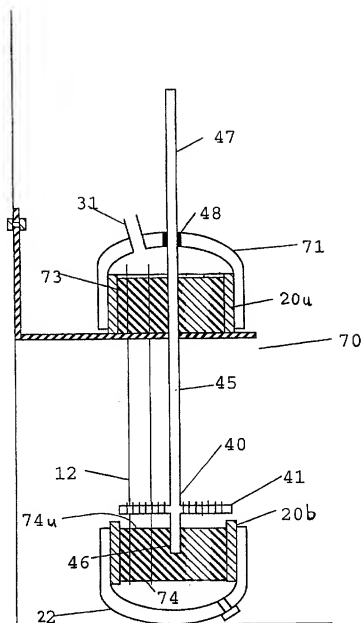


Fig. 7

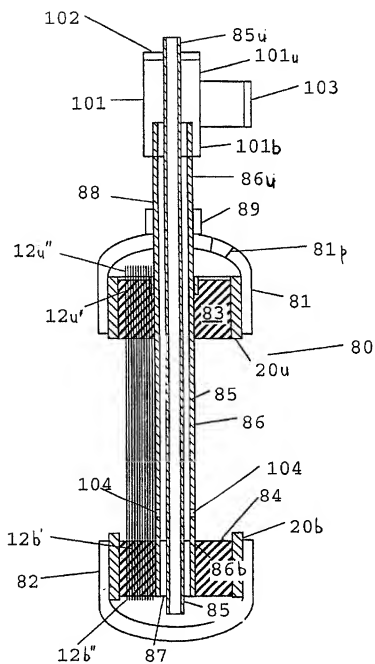


Fig. 8

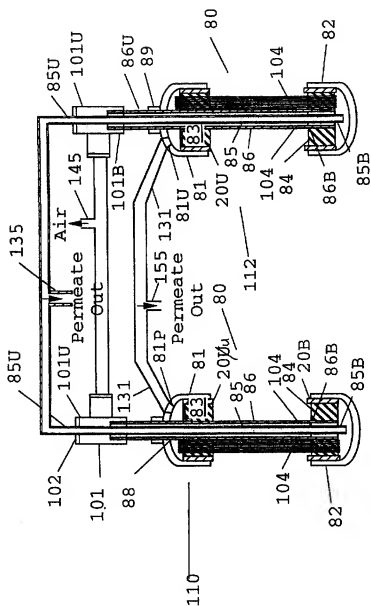


Fig. 9

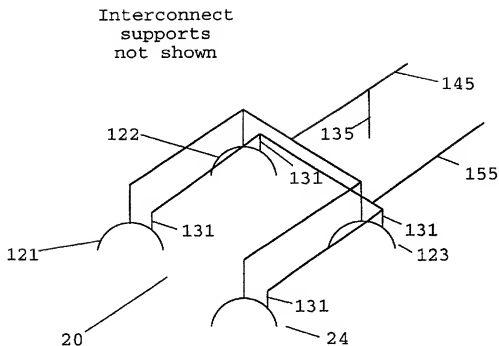


Fig. 10

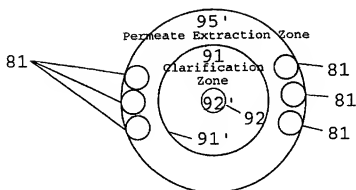


Fig. 12

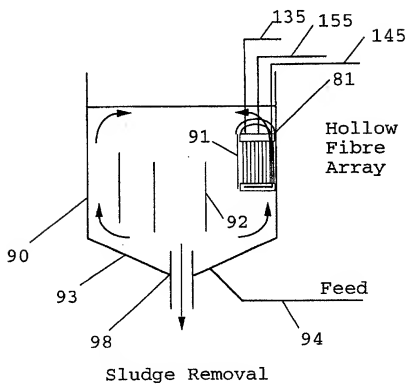


Fig. 11



FIGURE 13

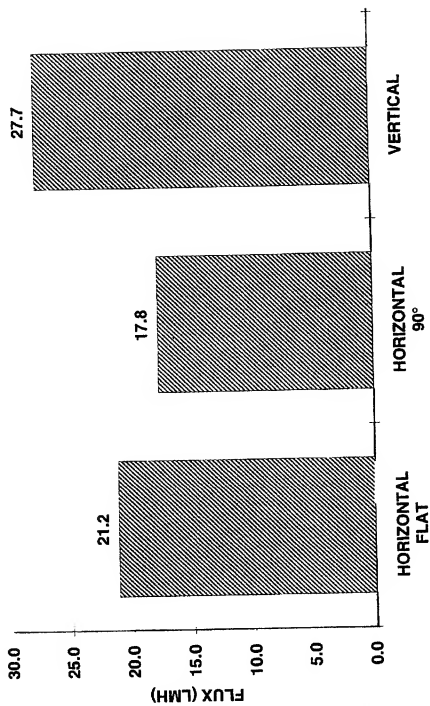


FIGURE 14

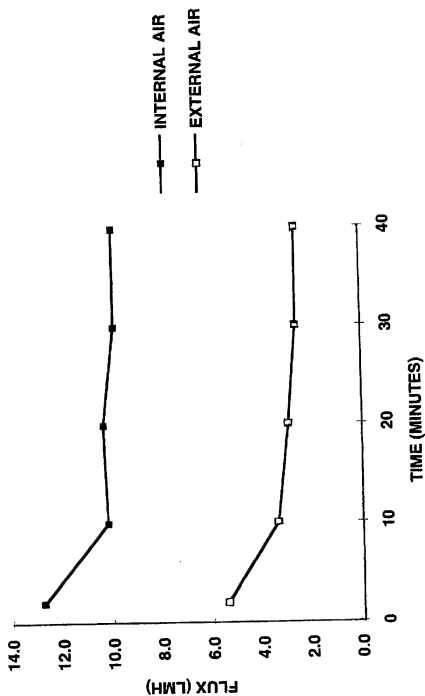
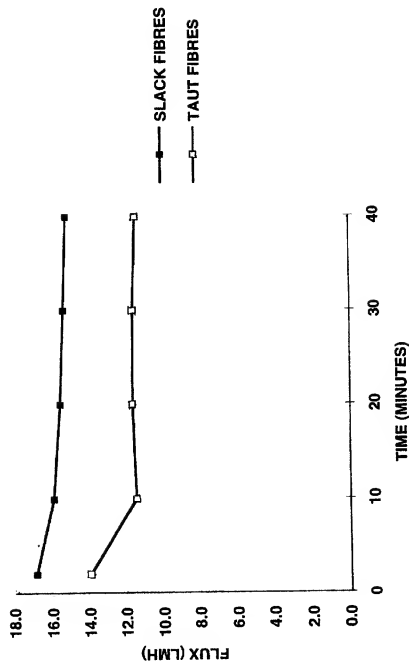


FIGURE 15



# **VERTICAL CYLINDRICAL SKEIN OF HOLLOW FIBER MEMBRANES AND METHOD OF MAINTAINING CLEAN FIBER SURFACES**

## **BACKGROUND OF THE INVENTION**

This is a continuation-in-part application of Ser. No. 08/514,119 filed Aug. 11, 1995 U.S. Pat. No. 5,639,373. Terms used in the parent case are summarized in a glossary herein, additional details in the parent case as well as in provisional application Ser. No. 60/012,921 filed Mar. 6, 1996, are incorporated herein by reference thereto as if fully set forth herein. In particular, considerations relative to the prior art and details of operation of prior art devices, all of which have been set forth in the '119 parent and provisional applications, are incorporated herein by reference thereto as if fully set forth herein.

This invention relates to a membrane device which is an improvement on a frameless array of hollow fiber membranes and a method of maintaining clean fiber surfaces while filtering a substrate to withdraw a permeate, which is also the subject of U.S. Pat. No. 5,248,424; and, to a method of forming a header for a skein of fibers.

This invention is particularly directed to relatively large systems for the microfiltration of liquids, and capitalizes on the simplicity and effectiveness of a configuration which dispenses with forming a module in which the fibers are confined. As in the '424 patent, the novel configuration efficiently uses air discharged near the base of a skein to produce bubbles in a specified size range, and in an amount large enough to scrub the fibers, and to provide controlled scrubbing of fibers one against another ("inter-fiber scrubbing"). Unlike in the '424 system the fibers in a skein are vertical and do not present an arcuate configuration above a horizontal plane through the horizontal center-line of a header. As a result, the path of the rising bubbles is generally parallel to the fibers and is not crossed by the fibers of a vertical skein. Yet the bubbles scrub the fibers.

The restrictedly swayable fibers, because of their defined length, do not get entangled, and do not abrade each other excessively, as is likely in the '424 array.

The side-to-side displacement of an intermediate portion of each fiber within the "zone of confinement" or "bubble zone" is restricted by the fiber's length. The defined length of the fibers herein minimizes (i) shearing forces where the upper fibers are held in the upper header, (ii) excessive rotation of the upper portion of the fibers, as well as (iii) excessive abrasion between fibers. Such swaying motion of a fiber with side-to-side displacement is distinct from vibration which occurs when a fiber is taut, that is, when the length of the potted fiber exposed to substrate is not longer than the distance between the opposed faces of upper and lower headers holding the fiber. Such vibration is induced by bubbles in a process for exfoliating and precipitating dense particles in U.S. Pat. No. 5,209,852 to Sanaoka et al. Unlike the fibers held in the module used in the '852 process, in our novel skein, there is essentially no tension on each fiber because the opposed faces of the headers are spaced apart at a distance less than the length of an individual fiber.

The use of an array of fibers in the direct treatment of activated sludge in a bioreactor, is described in an article titled "Direct Solid-Liquid Separation Using Hollow Fiber Membrane in an Activated Sludge Aeration Tank" by Kazuo Yamamoto et al in *Wat. Sci. Tech.*, Vol. 21, Brighton pp 43-54, 1989, and discussed in the '424 patent, the disclosure

of which is incorporated by reference thereto as if fully set forth herein. The relatively poor performance obtained by Yamamoto et al was mainly due to the fact that they did not realize the critical importance of maintaining flux by aerating a skein of fibers from within and beneath the skein. They did not realize the necessity of thoroughly scrubbing substantially the entire surfaces of the fibers by flowing bubbles through the skein to keep the fibers awash in bubbles. This requirement becomes more pronounced as the number of fibers in the skein increases.

Tests using the device of Yamamoto et al indicate that when the air is provided outside the skein the flux decreases much faster over a period of as little as 50 hr, confirming the results obtained by them. This is evident in FIG. 1 described in greater detail below, in which the graphs show results obtained by Yamamoto et al, and the '424 array, as well as those with a vertical skein in which the headers are rectangular, all three assemblies using essentially identical fibers, under essentially identical conditions.

The investigation of Yamamoto et al with downwardly suspended fibers was continued and recent developments were reported in an article titled "Organic Stabilization and Nitrogen Removal in Membrane Separation Bioreactor for Domestic Wastewater Treatment" by C. Chiemsaisri et al delivered in a talk to the Conference on Membrane Technology in Wastewater Management, in Cape Town, South Africa, Mar. 2-5, 1992, also discussed in the '424 patent. The fibers were suspended downwardly and highly turbulent flow of water in alternate directions, was essential.

It is evident that the disclosure in either the Yamamoto et al or the Chiemsaisri et al reference indicated that the flow of air across the surfaces of the suspended fibers did little or nothing to inhibit the attachment of microorganisms from the substrate.

Later, in European patent application 0 598 909 A1 filed by Yamamoto et al, they sought to avoid the problem of build-up on the fibers by "spreading the hollow fibers in the form of a flat sheet" (see page 4, lines 46-7) and there is no indication how the fibers would be maintained in a spread position in actual use. Further, each array is held in a "structural member for enclosing and supporting the fastening member" (see page 3, line 42, and lines 51-52) which is contrary to the concept of a frameless array. Their FIGS. 14, and 18 emphasize the horizontal configuration in which the array is used. To combat build-up FIG. 13 depicts how the fibers would trough when the array is taken out of the reservoir to be "vibrated" or shaken. A prior art module is illustrated in FIG. 16 showing both ends of each fiber potted in a cylindrical header, each fiber forming a loop, the looped ends being free. As the data in FIG. 17 shows, use of the prior art cylindrical module with looped ends freely movable in the substrate, was less effective than the frameless array with spread apart looped fibers shown in FIG. 1.

## **SUMMARY OF THE INVENTION**

It has been discovered that for no known reason, fibers which are more than 5% but less than 10% longer than the fixed distance between the opposed faces of the headers of a vertical skein, tend to shear off at the face; and those 10% longer tend to clump up in the bubble zone; and, that a gas-scrubbed vertical cylindrical skein of substantially concentrically disposed, restrictedly swayable fibers, provides an optimum configuration of fibers through which bubbles of a fiber-cleansing gas ("scrubbing gas") when flowed vertically upwards, parallel to and along the surfaces of the fibers. In a skein of densely packed fibers, bubbles in such

a configuration are more effective cleansing agents than bubbles which are intercepted by arcuate fibers crossing the path of the rising bubbles. Bubbles of an oxygen-containing gas to promote growth of microbes unexpectedly fails to build-up growth of microbes on the surfaces of swaying fibers because the surfaces are "vertically air-scrubbed". Deposits of animate and/or inanimate particles upon the surfaces of fibers are minimized when the restricted swaying fibers are kept awash in codirectionally rising bubbles which rise with sufficient velocity to exert a physical scrubbing force (momentum provides the energy) to keep the fibers substantially free of deleterious deposits. Thus, an unexpectedly high flux is maintained in fibers over each unit area the surface of the skin fibers over a long period.

In a "gas-scrubbed assembly" comprising a skin and a gas-distribution means, the skin preferably has a surface area which is at least  $>1 \text{ m}^2$ , and opposed spaced-apart ends of the fibers are secured in spaced-apart headers, so that the fibers, when deployed in the substrate, acquire a generally vertical cylindrical profile within the substrate and sway independently within the bubble zone defined by at least one column of bubbles. The length of fibers between opposed surfaces of headers from which they extend, is in a critical range from at least 0.1% (percent) longer than the distance separating those opposed faces, but less than 5% longer. Usually the length of fibers is less than 2% longer, and most typically, less than 1% longer, so that sway of the fibers is confined within a vertical zone of movement, the periphery of which zone is defined by side-to-side movement of outer skin fibers; and, the majority of these fibers move in a slightly larger zone than one defined by the projected area of one header upon the other. Though the distance between headers is fixed during operation, the distance is preferably adjustable to provide an optimum length of fibers, within the aforesaid ranges, between the headers.

Permeate may be withdrawn from only one, usually the upper permeate collection means (pan or end-cap), or, in skins of large surface area greater than  $200 \text{ m}^2$ , from both (upper and lower) pans or end-caps. Most preferably, air is introduced between skin fibers by an air-tube potted centrally axially within the upper end-cap, the air-tube supplying air to a sparger near the base of the skin fibers, and simultaneously providing a spacer means to position and space the lower end-cap the requisite distance from the upper end-cap. The sparger is part of a gas-supply means which supplies cleansing gas. The air-tube may be internally provided with a concentric permeate withdrawal tube axially extending to the permeate collection zone in the lower end-cap, and in open fluid communication with it, to withdraw permeate from both the upper and lower end-caps. Alternatively, the permeate withdrawal tube from the lower end-cap may be externally disposed so as to withdraw permeate from a passage in the lower portion of the end-cap, the tube being led outside the skin fibers, to communicate with the permeate withdrawal tube from the upper end-cap.

Preferably, for maximum utilization of space on a header, the fibers are deliberately set in a spiral pattern by rolling a large array into a spiral roll and potted each end of the spiral roll directly in a cylindrical resin-confining means. Such resin-confining means is typically a cylindrical end-cap such as is used for PVC pipe, or, an open-ended cylindrical ring. For use, each ring of the skin is, in turn, secured in an end-cap. Whether directly potted in an end-cap, or first in a ring, then secured in an end-cap, an integral header is formed. Since, a cylindrical skin in use, requires an end-cap to serve as an integral header, an end-cap integral header will be referred to hereafter as an "end-cap" for brevity.

#### BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and additional objects and advantages of the invention will best be understood by reference to the following detailed description, accompanied by schematic illustrations of preferred embodiments of the invention, in which illustrations like reference numerals refer to like elements, and in which:

FIG. 1 is a graph in which the variation of flux is plotted as a function of time, showing three curves for three runs made with three different arrays, in each case, using the same amount of air, the identical membranes and the same membrane surface area. The results obtained by Yamamoto et al are plotted as curve 2 (under conditions modified to give them the benefit of doubt as to the experimental procedure employed, as explained below); the flux obtained using the gas-scrubbed assembly of the '424 patent is shown as curve 1; and the flux obtained using a gas-scrubbed assembly of equal surface area is shown as curve 3. The headers of the gas-scrubbed assembly are rectangular parallelepipeds.

FIG. 2 is a cross-sectional view schematically illustrating a cylindrical skin having upper and lower end-cap integral headers in each of which is directly potted an array of fibers in a finished header sealed at its periphery to the wall of the end-cap without a gasket; permeate is withdrawn separately from the upper and lower headers and the draw from each combined in a permeate withdrawal manifold. Except for the lower end-cap resting on the floor of the tank, or otherwise supported in the substrate, the skin is unsupported during operation. By "unsupported" is meant "not supported except for spacer means to space the headers".

FIG. 2A is a bottom plan view of a potted array held as a roll in a fiber-setting form, before the end of the roll is potted in a ring, so as to form an integral header in which the pattern of fibers is spiral.

FIG. 2B is a bottom plan view of a series of potted cylindrical arrays referred to as "ring arrays" because the ends are secured in stiff cylindrical rings, the arrays being nested with each successive ring array being slid over the previous one. The nested rings are then potted in a resin-confining ring.

FIG. 2C is a bottom plan view of a series of planar arrays, the widths of each being chosen so that they may be stacked, chord-like (that is, as successive chords in the resin-confining ring) before the stack is potted in the ring.

FIGS. 3 and 3A are a cross-sectional view schematically illustrating a cylindrical skin and end-cap integral headers as in FIG. 2, except that permeate is withdrawn from only the upper header.

FIG. 4 is a perspective view of a single array, schematically illustrated, of a row of substantially coplanarly disposed parallel fibers secured near their opposed terminal ends between spaced apart cards. Typically, a single such array with a large number of fibers is rolled up before being sequentially potted.

FIG. 5 illustrates a side elevational view of a cylindrical skin of fibers, with the ends of the fibers on a rolled strip potted in a ring clamped to a panel coated with a release coating, to describe in detail how a finished header is formed.

FIG. 6 is a side elevational view schematically illustrating another embodiment of a cylindrical skin in which a conventionally formed header is held in a permeate pan and permeate is withdrawn from the lower end-cap into the upper end-cap through a rigid permeate tube inserted

through both the upper and lower headers. Terminal portions of a permeate connector tube are held in fluid-tight engagement with the upper and lower headers so that the permeate tube functions as a spacer means, and at the same time, as a support for the upper end-cap.

FIG. 7 is a side elevational view schematically illustrating a cylindrical skin in which a ring header is formed first. The ring header is then sealed into an end-cap. In addition to the permeate tube, a rigid air supply tube is inserted through the upper end-cap and upper header into the central portion of the skin, the lower portion of the air supply tube being potted in the lower header, thus functioning as a spacer means, and at the same time, as a support for the upper end-cap.

FIG. 8 illustratively shows another embodiment of the skin in which the permeate tube is concentrically disposed within the air supply tube, and both are potted, near their lower ends in the lower header. Ports in the lower end of the air supply tube provide air near the base of the skin fibers.

FIG. 9 is a perspective view schematically illustrating a pair of skins in a bank in which the upper headers are supported on brackets on the vertical wall of a tank and the lower headers rest on the floor. The skins in combination with a gas-distribution means form a "gas-scrubbing assembly" deployed within a substrate, with the fibers suspended essentially vertically in the substrate.

FIG. 10 is an elevational view schematically illustrating a "stand-alone" cluster of skins.

FIG. 11 is an elevational view schematically illustrating a bank of skins in which the wall of a bank is formed, showing the convenience of having all piping connections outside the liquid.

FIG. 12 is a plan view of the bioreactor shown in FIG. 11 showing how multiple banks of skins may be positioned around the circumference of the bioreactor to form a large permeate extraction zone while a clarification zone is formed in the central portion with the help of baffles.

FIG. 13 is a bar graph showing flux (liters per meter<sup>2</sup> per hour, LMH) as a function of the orientation of a skin.

FIG. 14 is a graph in which flux is plotted as a function of time during which a vertical cylindrical skin is aerated at a constant flow rate of air provided in one instance, by external aeration, and in another instance, by internal aeration.

FIG. 15 is a graph in which flux is plotted as a function of time for the same cylindrical skin used in two different embodiments achieved by adjusting the distance between the headers; the first embodiment having spaced apart conventionally at the maximum distance to provide taut fibers, and the other having headers spaced closer together to provide swayable fibers. During the test each vertical cylindrical skin is aerated at a constant flow rate of air.

#### DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

The Cylindrical Skin and the Arrays which form it

The cylindrical skins of this invention may be used in a liquid-liquid separation process of choice, and more generally, in various separation processes. The skin is specifically adapted for use in microfiltration processes used to remove large organic molecules, emulsified organic liquids and colloidal or suspended solids, usually from water. Typical applications are (i) in a membrane bioreactor, to produce permeate as purified water and recycle biomass; for (ii) filtration of secondary effluent from wastewater treatment, to remove suspended solids and pathogenic bac-

teria; (iii) clarification of aqueous streams including filtration of surface water to produce drinking water (removal of colloids, long chain carboxylic acids and pathogens); (iv) separation of a permeable liquid component in biotechnological broths; (v) dewatering of metal hydroxide sludges; and, (vi) filtration of oily wastewater, inter alia.

Typically the skin is configured so that all connections for fluids entering or leaving the skin are provided in the upper header. Permeate is most preferably withdrawn through a tube passing through the upper header whether (i) the lower header collects no permeate (as explained below); or (ii) permeate collects in both the upper and lower headers. The substantially circumferential geometry of the potted skin fibers is determined by a "fiber-setting form" used to set the fibers in a ring before they are potted. Instead of a single array rolled into a spiral, the fibers may be arranged in plural concentric arrays, or in plural flat arrays arranged chord-like in the potting ring. After potting, a star-shaped sparger or other shaped gas-distribution means is positioned near the base of the skin fibers. The skin preferably operates in a substrate held in a reservoir at atmospheric pressure or above in the range up to about 10 atm in a pressurized vessel, without being confined within the shell of a module.

One or more arrays are substantially concentrically potted about a central vertical axis in headers, the surfaces of which are in horizontal (x-y) planes. Instead of a single continuous array, plural arrays may be made and joined together, end-to-end successively, to form a much larger array which can be extended as it is rolled into a spiral roll.

Operation of the cylindrical skin is affected by (a) the fiber chosen, (b) the amount of air used, and (c) the substrate to be filtered. The goal is to filter a slow-moving or captive substrate in a large container under ambient or elevated pressure, but preferably under essentially ambient pressure, and to maximize the efficiency of a skin which does so (filters) practically and economically.

By operating at ambient pressure, mounting the integral headers of the skin within a reservoir of substrate, and by allowing the fibers restricted movement within the bubble zone in a substrate, we minimize damage to the fibers. Because a header secures at least 10, preferably from 50 to 50,000 fibers, each generally at least 0.5 m long, in a skin, it provides a high surface area for filtration of the substrate.

The Fibers and How they are Densely Packed

The fibers divide a reservoir into a "feed zone" and a withdrawal zone referred to as a "permeate zone". The feed of substrate is introduced externally (referred to as "outside-in" flow) of the fibers, and resolved into "permeate" and "concentrate" streams. The skin, or a bank of skins of this invention is most preferably used for microfiltration with "outside-in" flow. Though at least one skin is replaceably disposed in a small reservoir having a volume up to about 10 L (liters) and even up to about 100 L, or more, a bank of skins is preferably used in a relatively large reservoir having a volume in excess of 1000 L, such as a flowing stream, more typically a pond or tank. Most typically, a bank or plural banks with collection means for the permeate, are mounted in a large tank under atmospheric pressure, and permeate is withdrawn from the tank.

Where a bank or plural banks of skins are placed within a tank or bio-reactor, and no liquid other than the permeate leaves the tank, it is referred to as a "dead end tank". Alternatively, a bank or plural banks may be placed within a bioreactor, permeate removed, and sludge disposed of; or, in a tank or clarifier used in conjunction with a bioreactor, permeate removed, and sludge disposed of.

The fibers used to form the skin may be formed of any conventional membrane material provided the fibers are flexible and have an average pore cross sectional diameter for microfiltration, namely in the range from about 1000 Å to 10000 Å. Typically fibers range from 1 m to about 5 m long, depending upon the dimensions of the body of substrate (depth and width) in which the skin is deployed. Preferred fibers operate with a transmembrane pressure differential in the range from 7 kPa (1 psi)-69 kPa (10 psi) and are used under ambient pressure with the permeate withdrawn under gravity. The fibers are chosen with a view to perform their desired function, and the dimensions of the skin are determined by the geometry of the headers and length of the fibers. It is unnecessary to confine a skin in a modular shell, and a skin is not.

For hollow fiber membranes, the outside diameter of a fiber is at least 20 µm and may be as large as about 3 mm, typically being in the range from about 0.1 mm to 2 mm. The larger the outside diameter the less desirable the ratio of surface area per unit volume of fiber. The wall thickness of a fiber is at least 5 µm and may be as much as 1.2 mm, typically being in the range from about 15% to about 60% of the outside diameter of the fiber, most preferably from 0.5 mm to 1.2 mm.

The number of fibers in a single array is arbitrary, typically being in the range from about 1000 to about 10000 for commercial applications, and the preferred surface area for a skin is in the range from 10 m<sup>2</sup> to 100 m<sup>2</sup>. The center to center distance of adjacent fibers is preferably in the range from 1.2 (1.2d) to about 5 times (5d) the outside diameter 'd' of a fiber. Preferred center-to-center spacing is from about 1.5d to 2d. The packing density of fibers, that is, the number of fibers per unit area of header preferably ranges from 4 to 50 fibers/cm<sup>2</sup> depending upon the diameters of the fibers.

The particular method of securing the fibers in each of the headers is not narrowly critical, the choice depending upon the materials of the header and the fiber, and the cost of using a method other than potting. However, it is essential that each of the fibers be secured in fluid-tight relationship within each header to avoid contamination of permeate. This is effected by potting the fibers essentially vertically, in closely-spaced relationship, substantially concentrically.

Preferred fibers are made of organic polymers and ceramics, whether isotropic, or anisotropic, with a thin layer or "skin" on the outside surface of the fibers. Fibers may be made from braided yarn covered with a water-insoluble polymeric material such as those disclosed in U.S. Pat. No. 5,472,607. Preferred organic polymers for fibers are polysulfones, poly(styrenes), including styrene-containing copolymers such as acrylonitrile-styrene, butadiene-styrene and styrene-vinylbenzyl-halide copolymers, polycarbonates, cellulosic polymers, polypropylene, poly(vinyl chloride), poly(ethylene terephthalate), and the like disclosed in U.S. Pat. No. 4,230,463 the disclosure of which is incorporated by reference thereto as if fully set forth herein. Preferred ceramic fibers are made from alumina, by E. I. duPont de Nemours Co. and disclosed in U.S. Pat. No. 4,069,157. The Headers

One integral header of a skin is displaceable in any direction relative to the other, either longitudinally (x-axis) or transversely (y-axis), only prior to submerging the skin for operation. To use a skin, the headers are vertically spaced apart in parallel relationship within a reservoir, for example, by mounting one header above another against a vertical wall of the reservoir which functions as a spacer means. This is also true prior to spacing one header directly above another with other spacer means such as bars, rods,

struts, I-beams, channels, and the like, to assemble plural skins into a "bank or cluster of skins" ("bank" for brevity). After assembly into a bank, a segment intermediate the potted ends of each individual fiber is displaceable along either the x- or the y-axis, because the fibers are loosely held in the skin.

Because each integral header is preferably directly potted in a ring of suitable material from which the header of cured potting resin is not removed, no gasket is required (hence referred to as "gasketless") between the cured resin of the header and the inner periphery of the ring. When the integral header is adhesively secured in an end-cap to form a permeate-collection zone, again, no gasket is required, though one may be used if the integral header is to be disassembled.

The fixing material to fix the fibers in a finished header (or fixing lamina) is most preferably either a thermosetting or thermoplastic synthetic resinous material, optionally reinforced with glass fibers, boron or graphite fibers and the like. Thermoplastic materials may be crystalline, such as polyolefins, polyamides (nylon), polycarbonates and the like, semi-crystalline such as polyetherether ketone (PEEK), or substantially amorphous, such as poly(vinyl chloride) (PVC), polyurethane and the like. Thermosetting resins commonly include polyesters, polyacetals, polyethers, cast acrylics, thermosetting polyurethanes and epoxy resins. Most preferred as a "fixing" material (so termed because it fixes the locations of the fibers relative to each other) is one which when cured is substantially rigid in a thickness of about 2 cm, and referred to generically as a "plastic" because of its hardness. Such a plastic has a hardness in the range from about Shore D 30 to Rockwell R 110 and is selected from the group consisting of epoxy resins, phenolics, acrylics, polycarbonate, nylon, polystyrene, polypropylene and ultra-high molecular weight polyethylene (UHMWPE). Polyurethane such as is commercially available under the brand names Adiprene® from Uniroval Chemical Company and Airthane® from Air Products, and commercially available epoxy resins such as Epon 828 are excellent fixing materials.

The resulting membrane device comprises, (i) a vertical cylindrical skin of a multiplicity of restrictedly swappable fibers, together having a surface area in the range from 1 m<sup>2</sup> to 1000 m<sup>2</sup>, preferably from 10 m<sup>2</sup> to 100 m<sup>2</sup>, secured only in spaced-apart headers; and (ii) a gas-scrubbing means which produces a column of bubbles rising within and near the base of the skin, and engulfing the skin. Bubbles generated have an average diameter in the range from about 0.1 mm to about 25 mm, or even larger. A fluid component is selectively removed from the substrate.

#### The Gas-Scrubbed Assembly

A gas-scrubbed assembly comprises, (a) at least one skin, or a bank of gas-scrubbed cylindrical skins of fibers which separate a desired permeate from a large body of multicomponent substrate having finely divided particulate matter in the range from 0.1 µm-44 µm dispersed therein, (b) each skin comprising at least 20 fibers having upper and lower terminal portions potted spaced-apart, in upper and lower end-cap integral headers ("end-caps"), respectively, the fibers being restrictedly swappable in a bubble zone, and (c) a shaped gas-distribution means adapted to provide a profusion of vertically ascending bubbles in a column above and in close proximity to the upper face of the lower header, the length of the fibers being from at least 0.1% but less than 65% greater than the distance between the opposed faces of the headers. The shaped gas-distribution means has through-passages therein through which gas is flowed, continuously

or intermittently, at a flow rate which is proportional to the number of fibers. The flow rate is generally in the range from 0.47–14 cm<sup>3</sup>/sec per fiber (0.001–0.03 scfm/fiber) (standard ft<sup>3</sup> per minute per fiber), typically in the range from 1.4–4.2 cm<sup>3</sup>/sec/fiber (0.003–0.009 scfm/fiber). The surface area of the fibers is not used to define the amount of air used because the air travels substantially vertically along the length of each fiber.

The gas-scrubbed assembly is used (i) in combination with vertically adjustable spacer means for mounting the headers in vertically spaced apart relationship, in open fluid communication with (ii) collection means for collecting the permeate; means for withdrawing the permeate; and, (iii) sufficient air to generate enough bubbles flowing upwardly through the skin, between and parallel to the fibers so as to keep the surfaces of the fibers substantially free from deposits of live microorganisms as well as small inanimate particles which may be present in the substrate.

With surprisingly little cleansing gas discharged from a sparger disposed between fibers near their base, the specific flux at equilibrium is maintained over a long period, typically from 50 hr to 1500 hr. The sparger of a gas-distribution means is disposed adjacent the upper ("fore") face of the lower header to generate a column of rising bubbles within which column the fibers are awash in bubbles. A bank of skins may additionally be "gas-scrubbed" with one or more air-tubes disposed between the lower headers of adjacent skins, most preferably, also adjacent the outermost fibers of the first and last skins, so that for "n" headers there are "n+1" additional air-tubes. Each end-cap is preferably a commercially available synthetic resinous "dish" typically provided for the shell of a heat exchanger, or a "cap" for a pipe having a diameter about the same as the cylindrical skin to be formed. The upper and lower headers are cylindrical discs having the same diameter, and plural such skins may be clustered in a single row, or multiple rows, or in a honeycomb cluster, the upper headers being interconnected for support, and the lower headers supported on the floor of the reservoir. Even skins of different diameters may be clustered as described, if the headers are adequately intersupported in the substrate. Appropriately positioned and interconnected gas-tubes extend from a gas (air) manifold to service the bank, and an appropriate manifold is provided to withdraw permeate. The type of gas (air) manifold is not narrowly critical provided it delivers bubbles in a preferred size range from about 0.1 mm to 25 mm, measured within a distance of from 1 cm to 50 cm from the through-passages generating them.

#### Operation of the System

Operation of the system relies upon positioning at least one skin, preferably a bank, close to a source of sufficient air or gas to maintain a desirable flux, and, to enable permeate to be collected from at least one header. A desirable flux is obtained, and provides the appropriate transmembrane pressure differential of the fibers under operating process conditions.

The transmembrane pressure differential is preferably generated with a conventional non-vacuum pump if the transmembrane pressure differential is sufficiently low in the range from 0.7 kPa (0.1 psi) to 1.01 kPa (1 bar), provided the pump generates the requisite suction. A pump which generates minimal suction may be used if an adequate "liquid head" is provided between the surface of the substrate and the point at which permeate is withdrawn. Moreover, as explained in greater detail below, once the permeate flow is induced by a pump, the pump may not be necessary, the permeate continuing to flow under a "siphoning effect".

Clearly, operating with fibers subjected to a transmembrane pressure differential in the range up to 101 kPa (14.7 psi), a non-vacuum pump will provide adequate service in a reservoir which is not pressurized; and, in the range from 101 kPa to about 345 kPa (50 psi), by superatmospheric pressure generated by a high liquid head, or, by a pressurized reservoir.

A process for separating a permeate from a substrate while maintaining relatively clean surfaces of fibers in an array, comprises, submerging a skin of restrictedly swivable substantially vertical fibers within the substrate so that upper and lower end-caps of the skin are mounted one above the other with a multiplicity of fibers secured between cylindrical end-caps, the fibers having their opposed terminal portions potted in open fluid communication with at least one end-cap; the fibers operating under a transmembrane pressure differential in the range from about 0.7 kPa (0.1 psi) to about 345 kPa (50 psi), and a length from at least 0.1% to about 2% greater than the direct distance between the opposed upper and lower faces of cured resin in the end-caps, so as to present, when the fibers are deployed, a generally vertical cylindrical skin of substantially concentrically disposed fibers; maintaining an essentially constant flux substantially the same as the equilibrium flux initially obtained, indicating that the surfaces of the fibers are substantially free from further build-up of deposits once the equilibrium flux is attained; collecting the permeate; and, withdrawing the permeate.

The foregoing process may be used in the operation of an anaerobic or aerobic biological reactor which has been retrofitted with the membrane device of this invention. The anaerobic reactor is a closed vessel and the scrubbing gas is a molecular oxygen-free gas, such as nitrogen.

An aerobic biological reactor may be retrofitted with at least one gas-scrubbed bank of vertical cylindrical skins, each skin made with from 500 to 5000 fibers in the range from 1 m to 3 m long, in combination with a permeate collection means, to operate the reactor without being encumbered by the numerous restrictions and limitations imposed by a secondary clarification system.

Typically, there is no cross flow of substrate across the surface of the fibers in a "dead end" tank. If there is any flow of substrate through the skin in a dead end tank, the flow is due to aeration provided beneath the skin, or to such mechanical mixing as may be employed to maintain the solids in suspension. There is generally more flow and higher fluid velocity through the skin in a tank into which substrate is being continuously flowed, but the velocity of fluid across the fibers is generally too insignificant to deter growing microorganisms from attaching themselves, or suspended particles, e.g. microscopic siliceous particles, from being deposited on the surfaces of the fibers.

FIG. 1 presents the results of a comparison of three runs made, one using the teachings of Yamamoto in his '89 publication (curve 2), but using an aerator which introduced air from the side and directed it radially inwards, as is shown in Chiemchaisri et al. A second run (curve 3) uses the gas-scrubbed assembly of the '424 patent, and the third run (curve 3) uses a gas-scrubbed skin as described herein except that the headers were rectangular parallelepipeds. The specific flux obtained with an assembly of an inverted parabolic array with an air distributor means (Yamamoto et al.), as disclosed in *Wat. Sci. Tech.* Vol. 21, Brighton pp 43–54, 1989, and, the parabolic array by Cote et al in the '424 patent, are compared to the specific flux obtained with the vertical skin of this invention.



The comparison is for the three assemblies having fibers with nominal pore size 0.2  $\mu$ m with essentially identical bores and surface area in 80 L tanks filled with the same activated sludge substrate. The differences between the stated experiment of Yamamoto et al. and that of the '424 patent are of record in the '424 patent, and the conditions of the comparison are incorporated by reference thereto as if fully set forth herein. The vertical skin used herein differs from the '424 skin only in the vertical configuration of the 280 fibers, each of which was about 1% longer than the distance between the spaced apart headers during operation. The flow rate of air for the vertical skin is 1.4 m<sup>3</sup>/hr/m<sup>2</sup> using a coarse bubble diffuser.

It will be evident from FIG. 1 in which the specific flux, liters per meter<sup>2</sup> per hr per unit pressure (conventionally written as (Lmh/KPa)), is plotted as a function of operating time for the three assemblies, that the curve, identified as reference numeral 3 for the flux for the vertical skin, provides about the same specific flux as the parabolic skin, identified as reference numeral 1. As can be seen, each specific flux reaches an equilibrium condition within less than 50 hr, but after about 250 hr, it is seen that the specific flux for the inverted parabolic array keeps declining but the other two assemblies reach an equilibrium.

Referring to FIG. 2 there is schematically illustrated, in cross-sectional view a vertical cylindrical skin 10, comprising a pair of similar upper and lower end-caps 21 and 22 respectively which serve as cylindrical permeate collection pans. Skin fibers are unsupported and unattached one to another intermediate the headers; permeate is withdrawn separately from the upper and lower headers and the draw from each combined in a permeate withdrawal manifold. Each "end-cap" has a finished upper/lower header formed directly in it, upper header 23 being substantially identical to lower header 24. Each header is formed by potting fibers 12 in a potting resin such as a polyurethane or an epoxy of sufficient stiffness to hold and seal the fibers under the conditions of use. An end-cap was found especially convenient for making relatively small surface area skins because an end-cap for poly(vinyl chloride) "PVC" pipe serves as an excellent header and is commercially readily available; for large surface area skins, commercially available larger headers are provided by glass fiber reinforced end-caps for cylindrical tanks. Though the fibers 12 are not shown as close together as they would normally be, it is essential that the fibers are not in contact with each other, but that they be spaced apart by the cured resin between them. It is also essential that the potting resin adhere to and seal the lower portions 12' of each of the fibers against leakage of fluid under operating conditions of the skin. Visual confirmation of a seal is afforded by the peripheries of the fibers being sealed at the upper (fore) and lower (aft) faces 23a and 23b of the upper header 23, and the fore and aft faces 24a and 24b respectively of the lower header 24. A conventional finished header may be used in which the ends 12' of the fibers would be flush (in substantially the same plane) as the lower face 24b. In the best mode, though not visible through an opaque end-cap, the open ends 12' of the fibers protrude from the headers' lower (aft or bottom) face 24b.

The finished upper header 23 (fixing lamina) is left adhered to the periphery of the end-cap 21 when the fugitive lamina is removed through a bore 26 in the upper header; and analogously, the finished lower header 24 is left adhered to the periphery of the end-cap 22 when the fugitive lamina is removed through a bore 27. The bores 26 and 27 in the upper and lower end-caps have permeate withdrawal tubes 31 and 32, respectively, connected in fluid-tight engagement

therein. The permeate tubes 31 and 32, in turn, are connected to a permeate withdrawal manifold 30.

A detail of a sparger 40 is provided in FIG. 3A. The star-shaped sparger 40 having radially outwardly extending tubular arms 41 and a central supply stub 42, supplies air which is directed into the tubular arms and discharged into the substrate through air passages 43 in the walls of the arms. An air feed tube 44 connected to the central supply stub 42 provides air to the sparger 40. The lower end of the central stub 42 is provided with short projecting nipples 45 the inner ends of which are brazed to the stub. The outer ends of the nipples are threaded. The central stub and nipples are easy to insert into the center of the mass of skin fibers. When centrally positioned, arms 41 which are threaded at one end, are threadedly secured to the outer ends of the nipples.

As illustrated in FIG. 2, lower end-cap 22 rests on the floor F of a tank, near a vertical wall W to which is secured a vertical mounting strut 52 with appropriate fastening means such as a nut 53 and bolt 54. A U-shaped bracket 51 extends laterally from the base of the mounting strut 52. The arms of the U-shaped bracket support the periphery of upper end-cap 21, and to ensure that the end-cap stays in position, it is secured to the U-shaped bracket with a right angle bracket and fastening means (not shown). A slot in mounting strut 52 permits the U-shaped bracket to be raised or lowered so that the desired distance between the opposed faces 23b and 24a of the upper and lower headers respectively is less than the length of any potted fiber, measured between those faces, by a desired amount. Adjustability is particularly desirable if the length of the fibers tends to change during service.

Instead of withdrawing permeate through both tubes 31 and 32 it may be desirable to withdraw permeate from both the upper 21 and lower end-caps through only the upper tube 31. If it is, a permeate connector tube 33 (shown in phantom outline), is inserted within the mass of skin fibers 12 through the headers 23 and 24, connecting the permeate collection zone 29 in the lower end-cap in open fluid communication with the permeate collection zone 28 in the upper end-cap; and, bore 27 is plugged.

As illustrated in FIG. 3, in the event that withdrawal of permeate from the upper permeate collection zone 28 alone is sufficient, and it is unnecessary to withdraw permeate from both the upper and lower zones 28 and 29, the lower bore 27 of the lower end-cap 22 is simply plugged with a plug 25. Since, under such circumstances, it does not matter if the lower ends 12' of the fibers are plugged, and permeate collection zone 29 serves no essential function, the zone 29 may be filled with potting resin.

The step-wise procedure for forming an array to be potted in the novel header is described with respect to an array "A" illustrated in FIG. 4, as follows:

A desired number of fibers 12 are each cut to about the same length with a sharp blade so as to leave both opposed ends of each fiber with an essentially circular cross-section. The fibers are coplanarily disposed side-by-side in a linear array on a flexible planar support means such as strips or cards 15 and 16 which can be formed into a loose roll. Preferably strips of stiff paper are coated with an adhesive, e.g. a commercially available polyethylene hot-melt adhesive, so that the fibers are glued to the strips and opposed terminal portions 12' respectively of the fibers, extend beyond the strips. The strips securing the fibers extend over only the intermediate portions 12' of the fibers. Alternatively, to avoid gluing fibers to the strips, flexible strips of an elastomeric material such as a 50-90 Shore A polyurethane having pre-formed parallel spaced-apart

grooves therein into which the opposed ends of fibers may be snugly held, can be used.

Referring to FIG. 5 there is schematically illustrated the position of a spiral roll of an array of fibers which roll is secured with a rubber band 18 or other clamping means as it is held for potting in ring 20 which is clamped (not shown) tightly to a flat plate 14 so as to seal the periphery of ring 20 against the plate. The thickness of a strip and/or adhesive is sufficient to ensure that the fibers in successive layers of the roll are kept spaced apart. Preferably, this thickness is about the same as, or relatively smaller than the outside diameter of a fiber, preferably from about 0.5d to 1d thick, which becomes the spacing between adjacent outside surfaces of fibers in successive layers of the spiral. FIG. 2A illustrates the spiral pattern of openings in the ends 12" of the fibers, obtained when the spiral roll is potted in a potting ring 20.

In another embodiment, a series of successively larger diameter circular arrays may be formed, each a small predetermined amount larger than the preceding one, and the arrays secured, preferably adhesively, one to the next, near their upper and lower peripheries respectively to form a dense cylindrical mass of fibers. In such a mass of fibers, each array is secured both to a contiguous array having a next smaller diameter, as well as to a contiguous array having a next larger diameter, except for the innermost and outermost arrays which have the smallest and largest diameter, respectively. After the nested arrays are potted in ring 20, the resulting pattern of concentric circles formed by the open lower ends 12" of the fibers in the lower face 24b of the lower header is illustrated in FIG. 2B.

To make a skin with plural arrays arranged chord-like within a ring 20 or resin-confining means, plural planar arrays are formed on pairs of strips, each having a length corresponding to its position as a chord within a potting ring in which the skin fibers are to be potted. That is, each array is formed on strips of diminishing width, measured from the central array which is formed on a strip having a width slightly less than the inner diameter of the ring 20 in which the stack is to be potted. The arrays are stacked within the ring, the widest array corresponding in position to the diameter of the ring. For a chosen fiber, the larger the surface area required in a skin, the greater the number of fibers in each array, the bigger the diameter of the ring, and the wider each chord-like array. The plural arrays are preferably adhered one to the other by coating the surfaces of fibers with adhesive prior to placing a strip of the successive array on the fibers. Alternatively, the stacked arrays may be held with a rubber band before being inserted in the potting ring. The resulting chord-like pattern of the open lower ends 12" of the fibers in the lower face 24b of the lower header is illustrated in FIG. 2C. Ease of handling and the desired density of fibers per unit area of header will normally determine the choice of an embodiment for forming the potted skin.

Referring further to FIG. 5, the ring 20 serves the function of potting pan for forming the upper and lower headers. After the skin fibers are potted in finished headers, and the fibers checked for leaks so that any individual defective fiber may be plugged, the ring is snugly held in an end-cap (not shown) which serves as a permeate collection pan. The ring 20 may be adhesively secured in the end-cap or is held in fluid-tight engagement with it using a circumferential gasket. Whether the strips separating successive rows of fibers are to be retained will determine the depth L1 or L1' of fugitive header. The header of fixing material (thickness L1-L2) may have a cushioning layer (thickness L2-L3). If a gasketing lamina is desired, when the header is to be

secured above a permeate pan, a liquid gasketing material is poured and cured over the fugitive lamina to provide the gasketing lamina of thickness L1-L4.

The description of the method of forming a header is detailed in the '119 parent application, and in the '921 provisional application, which description is incorporated by reference thereto as if fully set forth herein.

The restricted swayability of the fibers generates some intermittent 'snapping' motion of the fibers which may break the potted fibers around their circumferences, at the interface of the fore face and substrate. To combat such damage, the fixing material is preferably chosen so as to provide adequate cushioning of the fibers at the interface. Such a material is typically an elastomer having a hardness in the range from 50 Shore A to about 20 Shore D.

Where a chosen fixing material is so hard as to cause the aforesaid damage, it is minimized by providing an additional lamina of material which is softer than the fixing lamina, to serve as a cushioning lamina. Such a cushioning lamina is formed integrally with the fixing lamina, by pouring cushioning liquid (so termed for its function when cured) over the fixing lamina to a desired depth sufficient to provide enough 'give' around the circumferences of the fibers to minimize the risk of shearing. Such cushioning liquid, when cured is rubbery, having a hardness in the range from about Shore A 30 to Shore D 20, and is preferably a polyurethane or silicone or other elastomeric material which will adhere to the fixing lamina. Upon removal of the fugitive lamina, the finished header thus formed has the combined thicknesses of the fixing lamina and the cushioning lamina, when the strips 15 are cut away.

As illustrated in FIGS. 2 and 3, a finished integral header may be directly formed in end-cap 21 and 22 into which permeate is to flow, thus solving the problem of sealing a conventionally formed and demolded header in a permeate pan.

Referring to FIG. 6 there is schematically illustrated a skin 60 with conventionally formed and demolded upper and lower headers 63 and 64 respectively potting the terminal portions of fibers 12. Each header is formed as described in U.S. Pat. No. 5,202,023. The ends of fibers in an array held in a spiral roll with a rubber band are dipped in resin or paint to prevent resin penetration into the bores of the fibers during the potting process. The ends of the roll are then placed in a mold and uncured resin added to saturate the ends of the fibers and fill the spaces between each individual fibers in the roll. The cured, molded ends are removed from the molds and the molded ends cut off (see, bridging cols 11 and 12).

Upper header 63 is placed against the lip 67 of a stainless steel permeate pan 61 and sealed in it with a peripheral gasket 65 placed circumferentially between the vertical wall of the header 23 and the vertical peripheral surface of the wall 66 of the permeate pan; alternatively, as illustrated in lower permeate pan 62, the gasket 65 may be placed between the lower peripheral surface of the lower header 64 and the peripheral lip 67 on which the header rests. A suitable sealing gasket or sealing compound typically used is a polyurethane or silicone resin. The periphery of each header is secured to its respective permeate pan with screws or other suitable fastening means to ensure a fluid-tight seal. The strips on which the array of fibers was held prior to being potted remain in the header, though not shown in the Figure.

As seen, the open ends of the embedded terminal portions 12' of the fibers are in the same plane as the lower face of the header 11 because the fibers are conventionally potted

and the header sectioned to expose the open ends. In this prior art method, sectioning the mold unavoidably damages at least some, and typically, a substantial number of the embedded fibers. Permeate connector tube is press-fitted when it is inserted in through-bore in the upper and lower headers after they are demolded and the plugged ends of the fibers cut away. As before, the skin is provided with a sparger 40 supplied by a flexible air supply tube 44 and the lower permeate pan 62 rests on floor F of a tank. The upper permeate pan rests on a U-shaped bracket 51 positioned so as to provide the desired slack in the fibers 12.

In the most preferred embodiment the novel method of potting disclosed herein is used. Because this method denies ready access to the ends of the fibers once finished headers are formed within a ring or an end-cap, the ends of the fibers protrude from the lower face 24b of the lower header 24 and the upper face 23a of the upper header 23 into the respective permeate collection zones.

Referring to FIG. 7 there is illustrated a skin 70 with upper and lower end-caps in which are sealed upper and lower ring headers formed in upper and lower rings 20a and 20b respectively, after the fibers in the skin are tested to determine if any is defective. Before an array is rolled into a spiral, as before, a sparger 40 with a rigid air-supply tube 45 is placed in the array so that upon forming a spiral roll the air-supply tube is centrally axially held within the roll. The lower end of the roll is then potted forming a lower finished header 74 in which the lower end 46 of the air-supply tube is potted, fixing the position of the arms 41 of the sparger just above the upper face 74a of the header 74.

In an analogous manner, an upper header 73 is formed in ring 20a and a permeate connector tube 33 press-fitted into aligned through-bore in the upper and lower headers. Upper end 47 of air-supply tube 45 is inserted through an axial bore 48 within upper end-cap 71 which is slipped over the ring 20a the outer periphery of which is coated with a suitable adhesive, to seal the ring 20a in the end-cap 71. The periphery of the upper end 47 is sealed in the end cap 71 with any conventional sealing compound.

Referring to FIG. 8 there is schematically illustrated another embodiment of a skin 80 in which rigid permeate tube 85 is held concentrically within a rigid air-supply tube 86 which is potted axially within skin fibers 12 held between opposed upper and lower headers 83 and 84 in upper and lower rings 20a and 20b which are in turn sealed in end-caps 81 and 82 respectively. For ease of manufacture, the lower end 85b of permeate tube 85 is snugly fitted and sealed in a bushing 87. The bushing 87 and end 85b are then inserted in the lower end 86b of the air supply tube 86 and sealed in it so that the annular zone between the outer surface of permeate tube 85 and the inner surface of air supply tube 86 will duct air to the base of the fibers but not permit permeate to enter the annular zone. The air supply tube is then placed on an array and the array is rolled into a spiral which is held at each end with rubber bands. The lower end of the roll is placed in a ring 20b and a lower ring header is formed with a finished header 84 as described above. It is preferred to use a relatively stiff elastomer having a hardness in the range from 50 Shore A to about 20 Shore D, and most preferred to use a polyurethane having a hardness in the range from 50 Shore A to about 20 Shore D, measured as set forth in ASTM D-790, such as PTU-921 available from Canadian Poly-Tech Systems. To form the upper finished header 83 the air supply tube is snugly inserted through an O-ring held in a central bore in a plate such as used in FIG. 5, to avoid loss of potting resin from the ring 20, and the fugitive resin and finishing resins poured

and cured, first one then the other, in the ring. Lower finished header 84 is formed with intermediate portions 12b' embedded, and terminal portions 12b'' protruding from the header's aft face. Upper finished header 83 is formed with intermediate portions 12a' embedded, and terminal portion 12a'' protruding from the header's fore face. After the finished headers 83 and 84 are formed and the fibers checked for defects, the upper end 86a of the air supply tube 86 is inserted through a central bore 88 in upper end-cap 81 and sealed within the bore with sealing compound or a collar 89. Preferably the permeate tube 85, the air supply tube 86 and the collar 89 are all made of PVC so that they are easily cemented together to make leak-proof connections.

As shown, permeate may be withdrawn through the permeate tube 85 from the permeate collection zone in the lower end-cap 82, and separately from the upper end-cap 81 through permeate withdrawal port 81p which may be threaded for attaching a pipe fitting. Alternatively, the permeate port 81p may be plugged and permeate withdrawn from both end-caps through the permeate tube 85.

Upper end 85a of permeate tube 85 and upper end 86a of air supply tube 86 are inserted through a T-fitting 101 through which air is supplied to the air supply tube 86. The lower end 101b of one of the arms of the T 101 is slip-fitted and sealed around the air supply tube. The upper end 101a of the other arm is inserted in a reducing bushing 102 and sealed around the permeate tube. Air supplied to intake 103 of the T 101 travels down the annular zone between the permeate tube and the air supply tube and exits through opposed ports 104 in the lower portion of the air supply tube, just above the upper face 84a of the lower header 84. It is preferred to thread ports 104 to threadably secure the ends of arms 41 to form a sparger which distributes air substantially uniformly across and above the surface 84a. Additional ports may be provided along the length of the vertical air supply tube, if desired.

Referring to FIG. 9 there is shown a bank 110 of a pair of side-by-side skins 111 and 112 substantially identical to skin 80 shown in FIG. 8, mounted in substrate against a wall W of a tank. The permeate withdrawal tubes 85 are manifolded to a common permeate manifold 135 and the T-fittings 101 for the air supply tubes 86 are manifolded to a common air supply 145. Permeate withdrawal tubes 131 are manifolded to a separate manifold 155 to provide greater flexibility than if manifolded with withdrawal tubes 85, and also to permit flushing the skin fibers. All connections to conduits to the bank are made to the upper end-caps for ease of operation. A skin with relatively low surface area may have as few as 100 fibers, while a skin with relatively large surface area may have as many as 2500 fibers, or more.

When permeate is withdrawn in the same place as the permeate withdrawal manifold, and the transmembrane pressure differential of the fibers is in the range from 35-75 kPa (5-10 psi), the manifold may be connected to the suction side of a centrifugal pump which will provide adequate NPSH.

In general, it is preferred to withdraw permeate from both the upper and lower headers, until the flux declines to so low a level as to require that the fibers be backwashed or backflushed. The skins may be backwashed by introducing a backwashing fluid through the permeate manifold under sufficient pressure to force the fluid through the pores of the membranes. This may be done in a skin having the configuration shown in FIGS. 2, 3, 6 and 7. The fibers may be backflushed in skins 111 and 112 in the same manner as the skin shown in FIG. 8. Backflushing fluid is introduced through one permeate tube (as for example one connected to

permeate port 81p) and removed through the other permeate tube (85). The skins 111 and 112 may also be backwashed by blocking flow of permeate through one manifold and pressuring backwashing fluid through the other permeate manifold.

Referring to FIG. 10, there is schematically illustrated another embodiment of an assembly, referred to as a "stand-alone" bank or cluster 120 of skins, four of which are referenced by numerals 121, 122, 123 and 124. The cluster is referred to as being a "stand-alone" because the spacer means between end-caps is provided by the concentric air-supply and permeate tubes potted in the headers. A cluster is usually used when mounting the skins against the wall of a reservoir is less effective than placing the cluster in spaced-apart relationship from a wall of the tank. In other respects, the cluster 120 is analogous to the wall-mounted bank 110 illustrated in FIG. 9. As will now be evident, the number of skins connected in a cluster is limited only by the connections provided to manifold the skins adequately.

In the best mode illustrated, each upper end-cap is provided with rigid PVC tubular nipples adapted to be coupled with fittings such as elbows and tees to the appropriate manifolds.

In another embodiment of the invention, a bioreactor is retrofitted with plural banks of skins schematically illustrated in the elevational view shown in FIG. 11, and the plan view shown in FIG. 12. The clarifier tank is a large circular tank 90 provided with a vertical, circular outer baffle 91, a vertical circular inner baffle 92, and a bottom 93 which slopes towards the center (apex) for drainage of accumulating sludge. Alternatively, the baffles may be individual, closely spaced rectangular plates arranged in outer and inner circles, but continuous cylindrical baffles (shown) are preferred. Irrespective of which baffles are used, the baffles are located so that their bottom peripheries are located at a chosen vertical distance above the bottom. Feed is introduced through feed line 94 in the bottom of the tank 90 until the level of the substrate rises above the outer baffle 91.

A bank 210 of plural side-by-side skins, analogous to those in the bank 110 depicted in FIG. 9, is deployed against the periphery of the inner wall of a bioreactor 90 with suitable mounting means in an outer annular permeate extraction zone 95 (FIG. 12) formed between the circular outer baffle 91 and the wall of the tank 90, at a depth sufficient to submerge the fibers. A clarification zone 91' is defined between the outer circular baffle 91 and inner circular baffle 92. The inner circular baffle 92 provides a vertical axial passage 92' through which substrate is fed into the tank 90. The side-by-side skins form a dense curtain of fibers hanging vertically between upper 81 and lower 82 end-caps. Permeate is withdrawn through permeate manifolds 135 and 155 and air is introduced through air-manifold 145, extending along the inner wall of the tank and branching out with connections to adjacent end-caps. Because air is sparged between fibers in such a manner as to have bubbles contact essentially the entire surface of each fiber which is continuously awash with bubbles while the fibers are vertical, the air is in contact with the surfaces of the fibers longer than if they were arcuate, and the air is used most effectively to maintain a high flux for a longer period of time than would otherwise be maintained.

It will be evident that if the tank is at ground level, there will be insufficient liquid head to induce a desirable liquid head under gravity alone. Without an adequate siphoning effect, a centrifugal pump may be used to produce the necessary suction. Such a pump should be capable of running dry for a short period, and of maintaining a vacuum

on the suction side of from 25.5 cm (10")-51 cm (20") of Hg. or -35 kPa (-5 psi) to -70 kPa (-10 psi). Examples of such pumps rated at 18.9 L/min (5 gpm) @ 15" Hg. are (i) flexible-impeller centrifugal pumps, e.g. Jabsco #30510-2003; (ii) air operated diaphragm pumps, e.g. Wilden M2; (iii) progressing cavity pumps, e.g. Ramco 3561; and (iv) hosepumps, e.g. Waukesha SP 25.

#### EXAMPLE 1

Microfiltration of an activated sludge at 30° C. having a concentration of 25 g/L (2.5% TSS) is carried out with a cylindrical skin of polysulfone fibers in a pilot plant tank. The fibers are "air scrubbed" at a flow rate of 12 CFM (0.34 m<sup>3</sup>/min) with a coarse bubble diffuser generating bubbles in the range from about 5 mm to 25 mm in nominal diameter. The air is sufficient not only for the oxidation requirements of the biomass but also for adequate scrubbing. The fibers have an outside diameter of 1.7 mm, a wall thickness of about 0.5 mm, and a surface porosity in the range from about 20% to 40% with pores about 0.2 µm in diameter, both latter physical properties being determined by a molecular weight cut off at 200,000 Daltons. The skin which has 1440 fibers with a surface area of 12 m<sup>2</sup> is wall-mounted in the tank, the vertical spaced apart distance of the headers being about 1% less than the length of a fiber in the skin. The opposed ends of the fibers are potted in upper and lower headers respectively, each about 41 cm long and 10 cm wide. The fixing material of the headers is a polyurethane having a hardness in the range from 50-90 Shore A. The average transmembrane pressure differential is about 34.5 kPa (5 psi). Permeate is withdrawn through a conduit connected to a pump generating about 34.5 kPa (5 psi) suction. Permeate is withdrawn at a specific flux of about 0.7 l/m<sup>2</sup>/kPa yielding about 4.8 l/min of permeate which has an average turbidity of <0.8 NTU, which is a turbidity not discernible to the naked eye.

#### EXAMPLE 2

Comparison of Operation of a Vertical Skin (ZW 72) in Different Orientations

In the following comparison, three pairs of identical skins with equally slack fibers are variously positioned (as designated) above aerators in a bioreactor. Each pair is subjected to the same discharge of air through identical aerators. Rectangular but not square headers are chosen to determine whether there is a difference between each of two flat horizontal orientations, which difference would not exist in a horizontal skin with cylindrical headers. A pair of identical rectangular skins, each having headers 41.66 cm (16.4 in) in length (x-axis), 10.16 cm (4 in) in width (y-axis) and 7.62 cm (3 in) in height (z-axis), in which are potted 1296 Zenon® MF200 microfiltration fibers presenting a nominal fiber surface area of 6.25 m<sup>2</sup>, were tested in three different orientations in a bioreactor treating domestic wastewaters. The fibers used are the same as those used in Example 1 above. The distance between opposed faces of headers is 90 cm (35.4 in) which is about 2% less than the length of each fiber potted in those headers.

In a first test, the two (first and second) skins were stacked laterally, each in the same direction along the longitudinal axis, with a 2.5 cm (1 in) thick spacer between the headers, the headers of each skin being in a horizontal flat orientation (area 41.66 cm×7.62 cm) is spaced apart 7.62 cm (3 in) above the floor on which lies the aerators in the form of three side-by-side linear tubes with 3 mm (0.125") openings. The first skin which is directly above the aerators is therefore referred to as the "lower skin".

In a second test, the same first and second skeins are each rotated 90° about the longitudinal x-axis and placed contiguously one beside the other. In this "horizontal 90° orientation" (area defined by 10.16 cm x 7.62 cm) is spaced apart from the aerators as in the prior test.

In a third test, the first and second skeins are placed side-by-side in vertical orientations and aeration is provided with a rectangular tube around the periphery of the skein, with perforations in the tube, and there is no internal aerator.

Each test provides the fibers in each orientation with the identical amount of air. Permeate was withdrawn with a pump with a NPSH of 0.3 bar (10" of Hg). The conditions were held constant until it was observed that the flux obtained for each test was substantially constant, this being the equilibrium value. After this occurred, each skein was back pulsed for 30 sec with permeate every 5 minutes to maintain the flux at the equilibrium value.

The test conditions for each of the above three runs were as follows:

TSS in bioreactor 8 g/L; Temperature of biomass 19° C. Flow rate of air 0.2124 m³/min/skein; Suction on fibers 25.4 cm of Hg

FIG. 13 is a bar graph which shows the average flux over a 24 hr period for each orientation of the skein as follows:

Orientation	Average flux LMH/hr over 24 hr
Horizontal flat	21.2 LMH
Horizontal 90°	17.8 LMH
Vertical	27.7 LMH

This conclusively demonstrates that the vertical orientation of the skein fibers produces the highest overall flux.

#### EXAMPLE 3

Comparison of Positions of Aerator Inside and Outside the Skein Fibers

In this test the difference in flux is measured in a bioreactor treating wastewater contaminated with ethylene glycol, the difference depending upon how a single cylindrical vertical skein (ZW 172) having a nominal surface area of 16 m² is aerated with 3.5 LMH (7.5 scfm). The skein is formed as shown in FIG. 16 around a central PVC pipe having an o.d. of 7.5 cm, the fibers being disposed in an annular zone around the central support, the radial width of the annular zone being about 7.5 cm, so that the o.d. of the skein is about 11.25 cm.

In a first test, air is introduced within the skein; in a second test, air is introduced around the periphery of the skein. After equilibrium is reached, operation is typically continued by back pulsing the skein with permeate at chosen intervals of time, the interval depending upon how quickly the fibers foul sufficiently to decrease the flux substantially.

The process conditions, which were held constant over the period of the test, were as follows:

TSS	17 g/L	Temperature of biomass	10.5° C.
Flow rate of air	0.2124 m³/min	Suction on fibers	25.4 cm of Hg

For external aeration

A perforated flexible tube with holes about 3 mm in diameter spaced about 2.5 cm apart was wrapped around the base of the ZW 72 skein and oriented so that air is discharged in a horizontal plane, so that bubbles enter laterally into the skein, between fibers. Thereafter the bubbles rise vertically through the skein fibers. Lateral discharge helps keep the holes from plugging prematurely.

For internal aeration

The central tubular support was used as the central air distribution manifold to duct air into five 4" lengths of 1/4" pipe with 1/8" holes at 1" intervals, plugged at one end, in open flow communication with the central pipe, forming a spoke-like sparger within the skein, at the base. The number of holes is about the same as the number in the external aerator, and the flow rate of air is the same. As before the holes discharge the air laterally within the skein, and the air bubbles rise vertically within the skein, and exit the skein below the upper header.

FIG. 14 is a plot of flux as a function of time, until the flux reaches an equilibrium value. Thereafter the flux may be maintained by back pulsing at regular intervals. As is evident, the equilibrium flux with external aeration is about 2.6 LMH, while the flux with internal aeration is about 9.9 LMH which is nearly a four-fold improvement. From the foregoing it will be evident that, since it is well-known that flux is a function of the flow rate of air, all other conditions being the same during normal operation, a higher flux is obtained with internal aeration with the same flow of air.

#### EXAMPLE 4

Comparison of skeins in which one has swayable fibers, the other does not

The slackness in the fibers is adjusted by decreasing the distance between headers. Essentially no slack is present (fibers are taut) when the headers are spaced at a distance which is the same as the length of a fiber between its opposed potted ends. A single ZW 72 skein is used having a nominal surface area of 6.7 m² is used in each test. In a bioreactor to treat wastewater contaminated with ethylene glycol. Aeration is provided as shown in FIG. 9 (no internal aeration) with lateral discharge of air bubbles into the skein fibers through which bubbles rose to the top.

In the first test the headers are vertically spaced apart so that the fibers are taut and could not sway.

In the second test, the headers were brought closer by 2 cm causing a 2.5% slackness in each fiber, permitting the slack fibers to sway.

As before the process conditions, which were held constant over the period of the test, were as follows:

Suspended solids	17 g/L	Temperature of biomass	10.5° C.
Flow rate of air	0.2124 m³/min	Suction on fibers	25.4 cm of Hg

FIG. 15 is a plot of flux as a function of time, until the flux reaches an equilibrium value. Thereafter the flux may be maintained by back pulsing at regular intervals as before in example 1. As is evident, the equilibrium flux with no swayability is about 11.5 LMH, while the flux with 2.5% slack is about 15.2 LMH, which is about a 30% improvement.

#### EXAMPLE 5

Filtration of water with a vertical cylindrical skein to obtain clarity

A cylindrical skein is constructed as in FIG. 16 with Zanon® MF2000 fibers 180 cm long, which provide a surface area of 25 m² in cylindrical headers having a diameter of 28 cm held in end-caps having an o.d. of 30 cm. Aeration is provided with a spider having perforated cross-arms with 3 mm (0.125") dia. openings which discharge about 10 liter/min (20 scfm, standard ft³/min) of air. This skein is used in four typical applications, the results of which are provided below. In each case, permeate is withdrawn with a centrifuge.

gal pump having a NPSH of about 0.3 bar ( $10^3$  Hg), and after equilibrium is reached, the skin is backflushed for 30 sec with permeate every 30 min.

A. Filtration of Surface (Pond) Water having 10 mg/L TSS Result—permeate having 0.0 mg/L TSS is withdrawn at a rate of 2000 liters/hr (LPH) with a turbidity of 0.1 NTU.

A "5 log" reduction (reduction of original concentration by five orders of magnitude) of bacteria, algae, giardia and cryptosporidium may be obtained, thus providing potable water.

B. Filtration of Raw Sewage with 100 mg/L TSS

Result—permeate having 0.0 mg/L suspended solids is withdrawn at a rate of 1000 LPH (liters/hr) with a turbidity of 0.2 NTU. Plural such skins may be used in a bank in the fully scale treatment of industrial wastewater.

C. Filtration of a mineral suspension containing 1000 mg/L TSS of iron oxide particles

Result—permeate having 0.0 mg/L suspended solids is withdrawn at a rate of 3000 LPH (liters/hr) with a turbidity of 0.1 NTU. High flux is maintained with industrial wastewater containing mineral particles.

D. Filtration of fermentation broth with 10,000 mg/L bacterial cells

Result—permeate having 0.0 mg/L suspended solids is withdrawn at a rate of 1000 LPH (liters/hr) with a turbidity of 0.1 NTU. The broth with a high biomass concentration is filtered non-destructively to yield the desired permeate, as well as to save living cells for reuse.

#### EXAMPLE 6

##### Special Purpose Mini-Skin

The following examples illustrate the use of a mini-skin for typical specific uses such as filtration of (i) raw sewage to obtain solids-free water samples for colorimetric analyses, (ii) surface water for use in a recreational vehicle ("camper") or motor home, or (iii) water from a small aquarium for fish or other marine animals.

A cylindrical mini-skin is constructed as shown in FIG. 16, with cylindrical headers having an o.d. of 5 cm (2") and a thickness of 2 cm (0.75") with 30 fibers, each 60 cm long to provide a surface area of 0.1 m<sup>2</sup>. The skin is mounted on a base on which is also removably disposed a blower to discharge 15 L/min of air at 12 kPa (3 psig) through a sparger which has 1.6 mm (0.0625") openings, the air flowing through the skin upwards along the fibers. Also removably mounted on the base is a peristaltic pump which produces a vacuum of 0.3 bar ( $10^3$  Hg). In each application, the self-contained skin with integral permeate pump and gas-discharge means, is placed, for operation, in a cylindrical container of the substrate to be filtered.

The results with each application (A)-(D) are listed below:

(i) Raw sewage contains 100 mg/L TSS; permeate containing 0.0 mg/L TSS having a turbidity of 0.2 NTU, is withdrawn at 0.1 LPH.

(ii) Aquarium water withdrawn contains 20 mg/L TSS, including algae, bacteria, fungus and fecal detritus; permeate containing 0.0 mg/L TSS having a turbidity of 0.2 NTU, is withdrawn at 0.1 LPH.

(iii) Pond water withdrawn contains 10 mg/L TSS; permeate containing 0.0 mg/L TSS having a turbidity of 0.2 NTU, is withdrawn at 0.1 LPH.

#### GLOSSARY

The following glossary is provided for terms in the approximate order in which they are used in the specification to define their meaning in the context in which they are used.

"array"—plural, essentially vertical fibers of substantially equal lengths, the one ends of each of which fibers are closely spaced-apart, either linearly in the transverse (y-axis herein) direction to provide at least one row, and typically plural rows of equidistantly spaced apart fibers. Less preferably, a multiplicity of fibers may be spaced in a random pattern. The opposed ends of fibers are sealed in opposed headers so that substrate does not contaminate permeate in permeate collection means in which the headers are peripherally sealed.

"bundle"—plural elements held together, e.g. plural arrays which may be a stack of planar arrays, or arcuate or circular arrays, or a rolled spiral.

"bank"—used for brevity, to refer to a bank of skins; in the bank, a row (or other configuration) of lower headers is directly beneath a row of upper headers.

"cylindrical skin"—a vertical skin in which the permeate collection means has a cylindrical configuration.

"dead end tank"—a tank or bioreactor from which no liquid other than the permeate is removed.

"fibers"—used for brevity to refer to hollow fiber membranes.

"flux"—unit flow (liters/hr), through a membrane of unit surface area (meter<sup>2</sup>), flux is given as Lm<sup>2</sup>/h or LMH.

"fugitive material"—material which is either (i) soluble in a medium in which the fibers and fixing material are not soluble, or (ii) fluidizable by virtue of having a melting point (if the material is crystalline) below that which might damage the fibers or fixing material; or, the material has a glass transition temperature T<sub>g</sub> (if the material is non-crystalline), below that which might damage the fibers or material(s) forming the non-fugitive header; or (iii) both soluble and fluidizable.

"header"—a solid body in which one of the terminal end portions of each one of a multiplicity of fibers in the skin, is sealingly secured to preclude substrate from contaminating the permeate in the lumens of the fibers. The body is of arbitrary dimensions formed from a natural or synthetic resinous material (thermoplastic or thermosetting).

"integral header"—combination of header and permeate collection means, in which combination the header is peripherally sealed in fluid-tight relationship with the permeate collection means.

"integral single skin"—a skin in an integral finished header is formed in the permeate pan or end-cap, sealing the header therein.

"mini-skin"—a self-contained gas-scrubbed assembly of a skin having a surface area less than about 5 m<sup>2</sup>, in combination with an integrally packaged gas blower and permeate pump.

"multicomponent liquid feed"—fruit juices to be clarified or concentrated; wastewater or water containing particulate matter; proteinaceous liquid dairy products such as cheese whey, and the like.

"non-vacuum pump"—generates a net section side pressure difference, or, net positive suction head (NPSH), adequate to provide the transmembrane pressure differential generated under the operating conditions; may be a centrifugal, rotary, crossflow, flow-through, or other type.

"permeability"—flux per unit pressure, Lm<sup>2</sup>/h/kPa; sometimes referred to as specific flux.

"permeate collection means"—receptacle beneath a header in which receptacle permeate collects.

"ring header"—header having a cylindrical shape.

"rectangular skien"—a vertical skien in which the permeate collection means has a configuration of a rectangular parallelepiped.

"skien"—used for brevity to refer to either a cylindrical skien or a vertical skien, or both, having plural arrays potted in opposed headers, the fibers having a critically defined length relative to the vertical distance between headers of the skien. The defined length limits the side-to-side movement of the fibers in the substrate in which they are deployed, except near the headers where there is negligible movement.

"skien fibers"—fibers which make up the cylindrical skien

"vertical skien"—an integrated combination of structural elements including (i) a multiplicity of vertical fibers of substantially equal length; (ii) a pair of headers in each of which are potted the opposed terminal portions of the fibers so as to leave their ends open; and, (iii) permeate collection means held peripherally in fluid-tight engagement with each header so as to collect permeate from the ends of the fibers.

"substrate"—multicomponent liquid feed.

"particulate matter"—micron-sized (from 1 to about 44  $\mu\text{m}$ ) and sub-micron sized (from about 0.1  $\mu\text{m}$  to 1  $\mu\text{m}$ ) filtrable matter which includes not only particulate inorganic matter, but also dead and live biologically active microorganisms, colloidal dispersions, solutions of large organic molecules such as fulvic acid and humic acid, and oil emulsions.

"restrictedly swayable"—the extent to which fibers may sway in a zone of confinement, which extent is determined by the free length of the fibers relative to the fixedly spaced-apart headers, and the turbulence of the substrate.

"stack of arrays"—plural rows of arrays, which are densely packed to form, after they are potted, a skien.

"substantially concentrically"—describes a configuration in which individual fibers are either vertical and spaced apart along the circumference of a circle drawn about the central vertical axis, or spirally disposed, successive layers of the fibers typically being closely spaced-apart in the x-y plane, not only radially outwards from the central axis, but also along the spiral in that plane so that they appear to be concentrically distributed at successively increasing radial distances from the central axis.

"transmembrane pressure differential"—pressure difference across a membrane wall, resulting from the process conditions under which the membrane is operating.

"unsupported"—not supported except for spacer means to space the headers.

"vacuum pump"—capable of generating a suction of at least 75 cm of Hg.

"zone of confinement" (or "bubble zone")—a zone through which bubbles rise along the outer surfaces of the fibers. The bubble zone, in turn, is determined by one or more columns of vertically rising gas bubbles generated near the base of a skien.

We claim:

1. In a gas-scrubbed assembly comprising, a microfiltration membrane device in combination with a gas-distribution means to minimize build-up of particulate deposits on the surfaces of hollow fiber membranes ("fibers") in said device, and to recover permeate from a multicomponent liquid substrate while leaving particulate matter therein, said membrane device comprising,

a multiplicity of fibers, unconfined in a shell of a module, said fibers being swayable in said substrate, said fibers

being subject to a transmembrane pressure differential in the range from about 0.7 kPa (0.1 psi) to about 345 kPa (50 psi);

a first and second header disposed in transversely spaced-apart relationship within said substrate, each header being formed with a potting resin cured in a resin-confining means;

said first header and second header having opposed terminal end portions of each fiber sealably secured therein, all open ends of said fibers extending from a permeate-discharging face of at least one header;

permeate collection means to collect said permeate through at least one of said headers sealably connected in open fluid communication with permeate-discharging faces of said headers;

means for withdrawing said permeate; and,

said gas-distribution means is located within a zone beneath said skien, said gas-distribution means having through-passages therein adapted to have sufficient gas flowed therethrough to generate enough bubbles flowing in a column of rising bubbles between and around said skien fibers, to keep surfaces of said fibers awash in bubbles;

said fibers, said headers and said permeate collection means together forming a vertical cylindrical skien wherein said fibers are essentially vertically disposed; said first header being upper and disposed in vertically spaced-apart relationship above said second header with opposed faces of said headers at a fixed distance, said fibers being substantially concentrically disposed relative to the vertical axis between said headers;

each of said fibers having substantially the same length, said length being from at least 0.1% greater, to less than 5% greater than said fixed distance so as to permit restricted displacement of an intermediate portion of each fiber, independently of the movement of another fiber;

the improvement comprising,

each said header having said fibers spaced apart by a flexible support means having a thickness corresponding to a desired lateral spacing between adjacent fibers, said support means extending over only each terminal portion of said fibers near their ends, so as to maintain said ends in closely-spaced apart relationship,

said gas distribution means being disposed between said fibers and having through-passages adapted to discharge said bubbles which rise vertically substantially parallel to, and in contact with said fibers, movement of which is restricted within said column; whereby said permeate is essentially continuously withdrawn.

2. The gas-scrubbed assembly of claim 1 wherein,

said restricted displacement is in the lateral or horizontal direction,

said headers are non-removably formed within said resin-confining means, and, said gas-distribution means includes an actuator means disposed adjacent to said lower header's upper face discharging said gas in an amount in the range from 0.47-14  $\text{cm}^3/\text{sec}$  per fiber (0.001  $\text{scfm}/\text{fiber}$  to about 0.03  $\text{scfm}/\text{fiber}$ ), said actuator means generating bubbles having an average diameter in the range from about 0.1 mm to about 25 mm, said bubbles maintain outer surfaces of said fibers essentially free from build-up of deposits of said particulate matter.

3. The gas-scrubbed assembly of claim 2 wherein, said gas-distribution means includes a vertical member centrally axially disposed within said skein and through at least one said header;
- said length is from 1% to less than 5% greater than said fixed distance;
- said fibers together have a surface area  $>1 \text{ m}^2$ , each fiber has a length  $>0.5$  meter;
- said fibers together have a surface area in the range from  $10$  to  $10^3 \text{ m}^2$ ;
- said headers are vertically adjustable to provide said fixed distance, and,
- said bubbles are in the size range from 1 mm to 25 mm measured in relatively close proximity, in the range from 1 cm to about 50 cm, to said through-passages.
4. The gas-scrubbed assembly of claim 2 wherein, each header includes both, a fiber-setting form to hold and set said fibers in a chosen pattern, and spacer means to maintain desired fiber-to-fiber spacing within said skein, said both being integral with said header;
- said fibers are potted within said synthetic resinous material to a depth in the range from 1 cm to about 5 cm and protrude through a permeate-discharging face of each said header in a range from about 0.1 mm to about 1 cm.
5. The gas-scrubbed assembly of claim 3 wherein, said permeate collection means includes a vertical member coaxially disposed within said gas distribution means' vertical member,
- said substrate is maintained at a pressure in the range from about 1–10 atm,
- said transmembrane pressure differential is in the range from 3.5 kPa (0.5 psi) to about 175 kPa (25 psi),
- opposed terminal end portions of said fibers are in open communication with each other through each said header;
- said fibers are in the range from 0.5 m to 5 m long, and, said terminal end portions of said fibers are potted within said mass of resin to a depth in the range from about 1 cm to about 5 cm.
6. The gas-scrubbed assembly of claim 5 wherein said particulate matter comprises biologically active microorganisms growing in said substrate.
7. The gas-scrubbed assembly of claim 5 wherein said particulate matter comprises finely divided inorganic particles.
8. The gas-scrubbed assembly of claim 1 wherein, each said fiber is formed from a material selected from the group consisting of natural and synthetic polymers, has an outside diameter in the range from about 20  $\mu\text{m}$  to about 3 mm, a wall thickness in the range from about 5  $\mu\text{m}$  to about 2 mm, and, a pore size in the range from 1000 Å to 10000 Å, each said header is a cylindrical disc having substantially the same dimensions, and, said gas is a molecular oxygen-containing gas.
9. In a microfiltration membrane device, for withdrawing permeate essentially continuously from a multicomponent liquid substrate, said membrane device including:
- a multiplicity of hollow fiber membranes, or fibers, unconfined in a shell of a module, said fibers being swayed in said substrate, said fibers being subject to a transmembrane pressure differential in the range from about 0.7 kPa (0.1 psi) to about 345 kPa (50 psi);
  - a first header and a second header disposed in transversely spaced-apart relationship with said second header within said substrate;

- said first header having a terminal end portion of each fiber secured therein, and said second header having an opposed terminal end portion of each fiber secured therein, all said fibers extending from a permeate-discharging face of at least one said header;
- said fibers being sealably secured with open ends of the fibers secured in fluid-tight relationship with each other in at least one of said headers;
- permeate collection means to collect said permeate through at least one of said headers sealably connected in open fluid communication with permeate-discharging faces of said headers;
- and, means for withdrawing said permeate;
- said fibers, said headers and said permeate collection means together forming a vertical cylindrical skein wherein said fibers are essentially vertically disposed;
- said first header being open and disposed in vertically spaced-apart relationship above said second header, with opposed faces at a fixed distance;
- each of said fibers having substantially the same length, said length being from 0.1% to less than 5% greater than said fixed distance so as to permit restricted displacement of an intermediate portion of each fiber, independently of the movement of another fiber;
- the improvement comprising,
- each said header having said fibers spaced apart by a flexible support means having a thickness corresponding to a desired lateral spacing between adjacent fibers, said support means extending over only each terminal portion of said fibers near their ends so as to maintain said ends in closely-spaced apart relationship.
10. The membrane device of claim 9 wherein, each said header is a mass of synthetic resinous material in which said terminal end portions are potted and said fibers are formed from natural or synthetic polymers; each said fiber has an outside diameter in the range from about 20  $\mu\text{m}$  to about 3 mm, a wall thickness in the range from about 5  $\mu\text{m}$  to about 2 mm, pore size in the range from 1,000 Å to 10,000 Å; and, said displacement is in the lateral or horizontal direction.
11. The membrane device of claim 10 wherein, said permeate collection means includes a vertical member axially disposed through said headers and within said skein,
- said substrate is maintained at a pressure in the range from about 1–10 atm, said fibers extend as a skein upwardly from a fiber-supporting face of each of said headers, each header has substantially the same dimensions, said fibers extend downwardly through the permeate-discharging face of said headers, and said permeate is discharged upwardly relative to said upper header.
12. The membrane device of claim 11 wherein, said fibers together have a surface area  $>1 \text{ m}^2$ , each fiber has a length  $>0.5$  meter, said fibers together have a surface area in the range from 10 to 103  $\text{m}^2$ , and, said terminal end portions of said fibers protrude through a permeate-discharging face of each said header in a range from about 0.1 mm to about 1 cm.
13. In a process for maintaining the outer surfaces of hollow fiber membranes essentially free from a build-up of deposits of particulate material while separating a permeate from a multicomponent liquid substrate in a reservoir, said process comprising,
- submerging skein fibers in an essentially vertical, cylindrical configuration within said substrate, said fibers



being unconfined in a modular shell, and securely held in vertically opposed, upper and lower headers spaced apart at a fixed distance, said fibers having substantially the same length and from at least 0.1% greater, to about 5% greater than said fixed distance, a transmembrane pressure differential in the range from about 0.7 kPa (0.1 psi) to about 345 kPa (50 psi), and length sufficiently greater than the direct distance between opposed faces of said first and second headers, so as to present said skein in a swayable configuration above a horizontal plane through the horizontal center-line of said lower header;

mounting said headers in fluid-tight open communication with collection means to collect said permeate;

flowing a fiber-cleaning gas through a gas-distribution means proximately disposed relative to said skein, within a zone beneath said skein, and contacting surfaces of said fibers with sufficient physical impact of bubbles of said gas to maintain essentially the entire length of each fibers in said skein awash with bubbles and essentially free from said build-up;

maintaining an essentially constant flux through said fibers substantially the same as an equilibrium flux initially obtained after commencing operation of said process;

collecting said permeate in said collection means; and, withdrawing said permeate.

the improvement comprising,

introducing said cleansing gas between said fibers within said skein to generate a column of said bubbles alongside and in contact with outer surfaces

of said fibers; said fibers spaced apart by a flexible support means having a thickness corresponding to a desired lateral spacing between adjacent fibers, said support means extending over only each terminal portion of said fibers near their ends, so as to maintain said ends in closely apart relationship;

restricting movement of said fibers to said vertical zone defined by lateral movement of outer fibers in said skein;

vertically gas-scrubbing said fibers outside surfaces with bubbles which flow upward in contact with said surfaces;

maintaining said surfaces substantially free from said deposits of particulate matter during a period when flux through said fibers has attained equilibrium; and simultaneously, essentially continuously, withdrawing said permeate.

14. The process of claim 13 wherein,

each said hollow fiber has an outside diameter in the range from about 20  $\mu\text{m}$  to about 3 mm, and a wall thickness in the range from about 5  $\mu\text{m}$  to about 1 mm;

said particulate matter is selected from the group consisting of microorganisms and finely divided inorganic particles; and,

said gas-distribution means discharges gas in an amount in the range from 0.47–14  $\text{cm}^3/\text{sec}$  per fiber (0.001 scfm/fiber to about 0.03 scfm/fiber) and generates bubbles having an average diameter in the range from about 1 mm to about 25 mm.

\* \* \* \* \*

UNITED STATES PATENT AND TRADEMARK OFFICE  
**CERTIFICATE OF CORRECTION**

PATENT NO. : 5,783,083

DATED : July 21, 1998

INVENTOR(S) : Wayne Jerald Henshaw et al.

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Claim 13, col. 28, line 6, delete "apart" and substitute -- spaced-apart --.

Signed and Sealed this  
Sixth Day of April, 1999



Q. TODD DICKINSON

Acting Commissioner of Patents and Trademarks

Attest:

Attesting Officer

COPY

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

*BOX: AMENDMENT (PATENTS)*

Applicants: Mahendran, Mailvaganam; Henshaw, Wayne J. et al.  
Application's Title: VERTICAL CYLINDRICAL SKEIN OF HOLLOW FIBER etc.  
Serial No.: 08/690,045  
Filed: July 31, 1996  
Group Art Unit: 3106 Examiner: ANA FORTUNA  
Docket No.: ZEN-9501-A

January 8, 1998

## AMENDMENT

Hon. Commissioner of Patents and Trademarks

Washington, D.C. 20231.

Sir:

This is in response to the office action mailed 10 September 1997. Please amend the above-identified patent application as follows:

## IN THE SPECIFICATION:

page 1, line 6, after "1995" insert now issued as U.S. Patent No. 5,639,273.

## IN THE CLAIMS:

Cancel claims 15 - 19 drawn to a method of making a header of hollow fibers classified in class 264, subclass Dig. 48, without prejudice, these claims having been withdrawn as a result of an election between Groups in a requirement for restriction.

1. (Amended) In a gas-scrubbed assembly comprising, a microfiltration membrane device in combination with a gas-distribution means to minimize build-up of particulate deposits on the surfaces of hollow fiber membranes ("fibers") in said device, and to recover permeate from a multicomponent liquid substrate while leaving particulate matter therein, said membrane device comprising,

a multiplicity of fibers, unconfined in a shell of a module, said fibers being swayable in said substrate, said fibers being subject to a transmembrane pressure differential in the range from about 0.7 kPa (0.1 psi) to about 345 kPa (50 psi);

a first and second header disposed in transversely spaced-apart relationship within said substrate, each header being formed with a potting resin cured in a resin-confining means;

said first header and second header having opposed terminal end portions of each fiber sealingly secured therein, all open ends of said fibers extending from a permeate-discharging face of at least one header;

permeate collection means to collect said permeate through at least one of said headers sealingly connected in open fluid communication with permeate-discharging faces of said headers;

means for withdrawing said permeate; and,

said gas-distribution means is located within a zone beneath said skein, said gas-distribution means having through-passages therein adapted to have sufficient gas flowed therethrough to generate enough bubbles flowing in a column of rising bubbles between and around said skein fibers, to keep surfaces of said fibers awash in bubbles;

[ the improvement comprising,]

said fibers, said headers and said permeate collection means together forming a vertical cylindrical skein wherein said fibers are essentially vertically disposed; said first header being upper and disposed in vertically spaced-apart relationship above said second header with opposed faces of said headers at a fixed distance, said fibers being substantially concentrically disposed relative to the vertical axis between said headers;

each of said fibers having substantially the same length, said length being from at least 0.1% greater, to less than 5% greater than said fixed distance so as to permit restricted displacement of an intermediate portion of each fiber, independently of the movement of another fiber; [ and, ]

the improvement comprising.

each said header having said fibers spaced apart by a flexible support means having a thickness corresponding to a desired lateral spacing between adjacent fibers, said support means extending over only each terminal portion of said fibers near their ends, so as to maintain said ends in closely-spaced apart relationship.

said gas distribution means being disposed between said fibers and having through-passages adapted to discharge said bubbles which rise vertically substantially parallel to, and in contact with said fibers, movement of which is restricted within said column;

whereby said permeate is essentially continuously withdrawn [ while concentration of said particulate matter in said substrate is increased ].

9. (Amended) In a microfiltration membrane device, for withdrawing permeate essentially continuously from a multicomponent liquid substrate [ while increasing the concentration of particulate material therein ], said membrane device including: a multiplicity of hollow fiber membranes, or fibers, unconfined in a shell of a module, said fibers being swayable in said substrate, said fibers being subject to a trans-membrane pressure differential in the range from about 0.7 kPa (0.1 psi) to about 345 kPa (50 psi); a first header and a second header disposed in transversely spaced-apart relationship with said second header within said substrate; said first header having a terminal end portion of each fiber secured therein, and said second header having an opposed terminal end portion of each fiber secured therein, all said fibers extending from a permeate-discharging face of at least one said header; said fibers being sealingly secured with open ends of the fibers secured in fluid-tight relationship with each other in at least one of said headers; permeate collection means to collect said permeate through at least one of said headers sealingly connected in open fluid communication with permeate-discharging faces of said headers; and, means for withdrawing said permeate; [ the improvement comprising,]

said fibers, said headers and said permeate collection means together forming a vertical cylindrical skein wherein said fibers are essentially vertically disposed; said first header being upper and disposed in vertically spaced-apart relationship above said second header, with opposed faces at a fixed distance; each of said fibers having substantially the same length, said length being from 0.1% to less than 5% greater than said fixed distance so as to permit restricted displacement of an intermediate portion of each fiber, independently of the movement of another fiber;

the improvement comprising,

each said header having said fibers spaced apart by a flexible support means having a thickness corresponding to a desired lateral spacing between adjacent fibers, said support means extending over only each terminal portion of said fibers near their ends, so as to maintain said ends in closely-spaced apart relationship.

13. (Amended) In a process for maintaining the outer surfaces of hollow fiber membranes essentially free from a build-up of deposits of particulate material while separating a permeate from a multicomponent liquid substrate in a reservoir, said process comprising, submerging skein fibers in an essentially vertical, cylindrical configuration within said substrate said fibers being unconfined in a modular shell, [ said fibers being ] and securely held in [ laterally ] vertically opposed, [ spaced-apart first and second ] upper and lower headers spaced-apart at a fixed distance, said fibers having substantially the same length and from at least 0.1% greater, to about 5% greater than said fixed distance, a transmembrane pressure differential in the range from about 0.7 kPa (0.1 psi) to about 345 kPa (50 psi), and a length sufficiently greater than the direct distance between opposed faces of said first and second headers, so as to present said skein in a swayable configuration above a horizontal plane through the horizontal center-line of [ a ] said lower header; mounting said headers in fluid-tight open communication with collection means to collect said permeate;

flowing a fiber-cleansing gas through a gas-distribution means proximately disposed relative to said skein, within a zone beneath said skein, and contacting surfaces of said fibers with sufficient physical impact of bubbles of said gas to maintain essentially the entire length of each fiber in said skein awash with bubbles and essentially free from said build-up;

maintaining an essentially constant flux through said fibers substantially the same as an equilibrium flux initially obtained after commencing operation of said process;

collecting said permeate in said collection means; and,

withdrawing said permeate,  
the improvement comprising,

[ deploying said skein fibers within said column in an essentially vertical, cylindrical configuration, with said headers in fixed spaced apart relationship at a fixed distance, said skein having fibers of substantially the same length and from at least 0.1% greater, to about 5% greater than said fixed distance, said fibers being independently swayable from side-to-side within a vertical zone of movement; ]

introducing said cleansing gas between said fibers within said skein to generate a column of said bubbles alongside and in contact with outer surfaces of said fibers; restricting movement of said fibers to said vertical zone defined by lateral movement of outer fibers in said skein;

vertically gas-scrubbing said fibers' outside surfaces with bubbles which flow upward in contact with said surfaces;

maintaining said surfaces substantially free from said deposits of particulate matter during a period when flux through said fibers has attained equilibrium; and, simultaneously, essentially continuously, withdrawing said permeate [ while increasing the concentration of said particulate material in said substrate ].

#### REMARKS

The provisional election of claims in Group I, namely claims 1-12 and 13-14 is affirmed.

The non-statutory double patenting rejection based on a judicially created

doctrine, is respectfully traversed. It is respectfully submitted that the terminal disclaimer filed herewith overcomes the rejection of record.

Each of the independent claims 1, 9 and 13 has been amended in view of the Japanese Patent Application No. Sho 61 [1986] - 292045 of inventor Tamiro Kunio which was cited by an European examiner. The reference discloses a skein having fibers which are deliberately longer than in a conventional skein, as are the fibers in applicants' invention. Such a skein with fibers longer than in a conventional skein is referred to herein as an "extended skein".

Kunio states "Conventionally, it (the degree of excess length) has been set with excess length of approximately 5 percent." (see sentence bridging pages 3 and 4, pagination numerals appear at the bottom of the pages) indicating that, in Japan, it was conventional to set the excess length of fibers in a vertical skein at about 5%. Kunio discovered that if the excess length was set for 1% to 4%, an extended vertical skein can withstand the rigors of backwashing much better than with other excess lengths either less than 1% or greater than 4%. The conventional system described by Kunio is a deadend filtration system, and since in such a system there is no provision for aerating a skein while filtering the substrate, neither Kunio nor others in Japan who used such a conventional system could have aerated their skeins during filtration.

As stated by Kunio "However, situations in which the multiple pieces of hollow yarn 2a become twisted then bent and damage (*sic*) have occurred as filtration and backwashing were repeated." (see lines 1 -3, top of page 3) indicating that each separate cycle of filtration and backwashing resulted in some damage. This is not surprising since backwashing is carried out with pressurized air blown through the lumens (longitudinal axial bores) of the fibers to dislodge deposits from the surfaces of the fibers, and simultaneously air is also blown around the outer surfaces of those fibers to make sure the dislodged deposits do not accumulate in the bundled fibers of the Kunio skein. Aeration of the outer surfaces is referred to by Kunio as a "bubbling operation" (see page 3, line 7 of the full paragraph in the middle of the page). However, even with 1% "slack", Kunio observed some damage with each backwashing cycle as graphically illustrated in his Fig 3. Such damage is unacceptable in a



commercially operated skein. In his Declaration under 37 C.F.R. 1.132, appended hereto, Steven Pedersen states "In a commercial installation, if backwashing damages only one (1) fiber out of one thousand (1000) fibers in a skein, the backwashing would be deemed unsatisfactory." (see numbered paragraph 10).

Applicants discovered that if the fibers in an extended skein were not bundled, but potted in spaced-apart relationship, spaced apart by a flexible support means, there was no damage to the fibers over an extended period. Applicants discovered that with spaced-apart fibers in headers of an extended skein, in which skein the specified "slack" may be in a range wider than the range Kunio required for survival of his skein during backwashing, skein fibers will be immune to the damage suffered by Kunio's skein fibers, provided the ends of the fibers were potted in spaced-apart relationship in a header which was not rigid. As explained herebelow, there is **no provision in Kunio** for aerating his skeins during filtration, and it is clear he never intended to do so. It is self-evident (and affirmed by Kenneth Goodboy in his appended Declaration under 37 C.F.R. 1.132, numbered paragraph 8) that if the equipment Kunio used was incapable of disposing of air used for aerating the skein while filtering, Kunio could not suggest aerating the skein **while filtering**.

Accordingly, claim 9 has been amended to specify the first essential limitation ("slack") in the preamble, and to define the second essential element. This second essential element is the "flexible support means having a thickness corresponding to a desired lateral spacing between adjacent fibers" in the headers. Claim 1 is directed to the skein of amended claim 9 in combination with gas distribution (typically, aeration) means. Claim 13 is specifically directed to vertically gas-scrubbing the biers' outer surfaces while maintaining "clean" fibers and **simultaneously filtering** the substrate.

Enclosed herewith is the Japanese text of the Japanese Patent Application; also enclosed is a certified translation of the Japanese text.

Referring to the "Conventional Art" Kunio states:

In the aforesaid configuration, when the differential pressure before and after the hollow yarn membrane filter 2 rises due to filtra-

tion and reaches a specified value, *a backwash operation is executed to perform an operation to wash off the solid portion which has adhered to the surfaces of the respective pieces of hollow yarn 2a. That is, a pressurized gas for backwashing is supplied inside the respective pieces of hollow yarn 2a of the hollow yarn membrane filter 2 via the aforesaid processing fluid discharge pipe 11. Simultaneously, a bubbling operation is executed from below the hollow yarn membrane filter 2. That is, a bubbling pipe 15 is arranged below the hollow yarn membrane filter 2 within the aforesaid container main unit 1, and bubble holes 16 are formed in the lower surface of this bubbling pipe 15. The aforesaid bubbling pipe 15 is connected to an air supply pipe 17 which has a shut-off valve 18. By supplying air to the aforesaid bubbling pipe 15 via the aforesaid air supply pipe 17, bubbles are generated from the aforesaid bubble holes 16. The hollow yarn membrane filter 2 is subject to bubbling by the aforesaid bubbles to improve the washing effect. An overflow pipe 19 is connected to the container main unit 1 so that it is positioned below the aforesaid diaphragm 3, and a shut-off valve 20 is positioned along said overflow pipe 19. Callout 21 in the diagram is a protecting tube, and this protecting tube 21 which allows the bubbles from the aforesaid bubbling to be effectively introduced into the hollow yarn membrane filter 2. (see middle of page 3, numbered at the bottom; italics supplied).*

Kunio goes on to describe what happens in conventional membrane filters when they are backwashed, and that the source of the **problems in backwashing** is due to the

"question of what degree of excess length should be set for the length (L1; a value larger than L2, since there is some looseness in the gap which is the aforesaid L2) of the hollow yarn 2a arranged between the two ends with respect to the distance . . . in order to effectively perform the aforesaid bubbling and prevent damage to the hollow yarn 2a has not been taken into account." (see bottom of page 3).

Kunio then states that "multiple pieces of hollow yarn 2a become twisted then bent and damage have (*sic*) occurred as filtration and backwashing were repeated." (see top of page 4), and offers the reason for such damage:

"This is thought to be because the hollow yarn 2a consists of a polymer-

ic material, and its specific gravity is almost equal to that of water, which is the main constituent of the processed fluid, so the hollow yarn 2a whirls up, then bends and becomes damaged." (see lines 3-6, page 4).

Finally Kunio offers a solution to the breakage problem during backwashing, namely, that "the excess length, which has been set to approximately 5 percent as mentioned above, may be shortened or eliminated." Kunio then states that the offered solution is not effective because "the following problems occur when such a method is adopted." (see middle of page 4). He then goes on to list the problems of the conventional backwashing which he has described, numbering them 1 through 3.

These problems may be summarized as follows:

- 1) greater restriction of excess length provides an insufficient bubbling effect;
- 2) in a dense bundle, flow to the fibers in the interior of the bundle is blocked;
- 3) solid material dropped off accumulates in the bundle.

Kunio reiterates the problem he has already addressed in the earlier portion of his specification, to emphasize it. He now puts it under the heading: "Problems To Be Solved By the Invention". Simply stated, his invention is designed to take into account how to determine excess length, the objective being "to provide a membrane filter which makes it possible to perform effective backwashing while preventing damage to the hollow yarn." (see lower portion of page 4).

Under the heading "Configuration of the Invention" Kunio lists the following subheadings: "Means To Solve Problems"; "Action"; "Embodiments"; and "Benefits of the Invention".

Under "Means To Solve Problems" Kunio states that he uses fibers which satisfy the following criterion:

$$0.01 \leq (\Delta L/L1) \leq 0.04$$

where, L1 represents the length of the hollow yarn arranged between the two bonding agent filling sections; L2 represents the gap between the two bonding agent filling sections; and,  $\Delta L = (L1 - L2)$

Under "Action" Kunio states that setting the excess length according to the foregoing criterion effectively solves such problems as (a) the drop in the backwashing effect which occurs due to the excess length being too small, and (b) the bending

and damage which result from the twisting of the hollow yarn which occurs due to the excess length being too great. (see middle of page 7).

Under "Embodiments" Kunio describes the vertical skein which he conventionally potted in two "bonding agent filling sections 6 secured with bundle securing members 7" (see lines 3-4 of page 3). Clearly the fibers are conventionally bundled and potted "by bundling many pieces of the hollow yarn and hardening both ends with resin, which is a bonding agent." (see first paragraph under "Conventional Art" on page 2). The bundle of fibers is secured by "a bundle securing member [which] is installed and secured at the outer circumference of the bonding agent filling sections filled with the aforesaid bonding agent, . . ." (see page 2, lines 1 and 2). In greater detail, Kunio describes how the bundle of fibers is held, as follows: "The aforesaid hollow yarn membrane filter 2 has a structure whereby multiple pieces of hollow yarn 2a are bundled at the outer circumference of a support member 4, and their upper and lower ends are secured by bonding agent filling sections 6, and, in addition, bundle securing members 7 are installed and secured from the outer circumferences thereof." (see sentence bridging bottom of page 2 and top of page 3 and Fig 1 identifying the cylindrical bundle securing ring 7). Such a method allows one to form a bundle with the maximum number of fibers within a cylindrical header, thus providing maximum filtration area for the number of fibers used in the bundle. However, the problem with conventional "bundling" is that too many fibers are in contact with one another with varying degrees of 'tightness', more specifically, fluid-tightness. The result is that when the bundle of fibers is potted, resin impregnates the bundle in direct proportion with the degree of fluid-tight contact between contiguous fibers. Where no resin is cured between fibers there is a likelihood that there will be fiber-to-fiber abrasion, and since the fibers are compressed (the walls of fibers are compressible), that the header holding the bundle will leak. The proclivity to leak is exacerbated when the fibers are of polymer and therefore radially compressible. In operation, these fibers which are contiguously bundled (touching each other in the bundle), chafe against each other as they sway. Such chafing simultaneously weakens the walls of the fibers and increases the likelihood of leakage of substrate between

them, the substrate getting into the permeate collection system. Note that Kunio states:

"2) When the hollow yarn membrane filter 2 is bundled in the aforesaid way in a condition in which multiple pieces of hollow yarn 2a are densely arranged, . . ." (see page 4, lines 12-13)

Applicants discovered how to solve the problem by **avoiding bundling** the fibers in contact with one and another. They sacrificed density of fibers to obtain better longevity (no fiber-to-fiber abrasion) and a leak-proof header. They did so by supporting spaced-apart fibers on a flexible support of desired thickness before potting the fibers, thus assuring a chosen spacing (specified by the center-to-center dimensions) between fibers in the header, and assuring the penetration of resin between every fiber. This additional limitation has been introduced into the amended article claims to the skein.

In a typical laboratory experiment, run over only as long (or short) a period as is required to provide data sought, the leakage of a header is not easily discovered unless one checks for permeate quality. In a commercially used membrane filter which is run over a long period, permeate quality is typically checked continually. Leakage of substrate through a header is a problem of the greatest concern because it results in contaminated permeate, the expenditure of great effort to discover just where the leak(s) has occurred, and quickly "trashing" a suspected skein. It is therefore of over-riding importance that the fibers in a skein be sealed in spaced apart relationship to one another, and applicants discovered how to do this.

There is no argument that Kunio discovered that there was a critical excess length which allowed hollow fibers in a vertical skein to survive the rigors of his **backwashing** process. Applicants discovery of the benefits of the extended skein were directed to a different process - **filtration**. Moreover, Kunio failed to find the leakage problem and therefore never sought a solution to that serious problem.

Since Kunio's data were generated only during backwashing, the excess length (of fibers) he decided was necessary for that operation was in the range from 1% to 4%. Since applicants were only concerned with filtration, not backwashing, the excess

they decided was necessary for filtration was in the range from at least 0.1% greater, to less than 5% greater than the fixed distance between faces of opposed headers. Since the range of 1% to 4% is within the range from 0.1% but less than 5%, the latter range is recited in the preamble of the claims.

Claims 1 - 8, and 9 - 12 (as were claims 13-14), were written in Jepson form, specifically because of applicant's U.S. Patent No. 5,39,373. Claim 9, as amended, recites the excess length as being a conventional aspect of the cylindrical vertical skein, as taught by Kunio, and now specifically identifies the unique relationship of spaced-apart fibers in the headers. The basis for "closely-spaced relationship" is found in the specification at page 12, line 7. The basis for "flexible support means" is found in the specification at page 21, line 8, and in original claim 15 (line 4).

Kunio's only concern was damage during backwashing during which "a pressurized gas for backwashing is supplied inside the . . . hollow yarn . . . Simultaneously, a bubbling operation is executed from below . . ." (see middle of page 3). Obviously, one cannot filter while backwashing because the pressure differential is in the wrong direction. Kunio never suggested modifying his equipment for filtering while, at the same time, bubbling a column of cleansing gas around the fibers of the skein. Moreover there is nothing in Kunio's teaching to motivate one skilled in the art to filter, and at the same time, bubble a cleansing gas between the fibers without passing pressurized gas inside the fibers. From a practical point of view, backwashing and filtration are entirely separate operations. One only considers backwashing when filtration is no longer satisfactory. No one of ordinary skill in the art considers backwashing while filtering because the pressure drop is in the wrong direction.

Note further that, though there is no teaching by Kunio as to the details of how backwashing is to be carried out, it is reasonable to expect that backwashing Kunio's skein requires using air under relatively high pressure, and that the air is pulsed (typically 5 second pressures) in accordance with a technique described in examples 3 and 4 of U.S. Patent No. 4,767,539 to Ford. In examples 3-10 air at 500 kPa (72.5 psig) is used; and in example 11, air is used at 475 kPa. The high pressure is required to overcome the transmembrane differential so that air being forced

through the pores of the membrane will help dislodge solids adhering to the outer surfaces of the fibers. Kunio obtains additional assistance to shed adhering solids by providing oscillating fibers. Kunio states: "As shown in Figure 2, this is because it is necessary for the hollow yarn 2a to oscillate to a certain extent when bubbling is performed during backwashing, and the solid portion gets shaken off by said oscillation." (see middle of page 6, middle of second full paragraph). The resulting effects of the combination of the foregoing "through-the-fiber" air, coupled with oscillation of the fibers during backwashing, is referred to in the detailed description quoted above, relevant portions of which are italicized.

Concluding his description of the extended length skein, Kunio states three (enumerated 1 to 3) benefits obtained during backwashing his skein, and points out that as a result of the foregoing three benefits, a fourth benefit is that such backwashing allows fibers near the center of the bundle to filter effectively. (see bottom of page 6, and top of page 7).

Kunio then summarizes the "Benefits of the Inventions" as follows: "As explained in detail above, through the hollow yarn membrane filter resulting from the present invention, there are great benefits in that it is possible to prevent the situation whereby the hollow yarn whirls up and therefore becomes twisted and bent or damaged and to provide effective backwashing."

There can be little doubt that Kunio's only concern was to provide an extended length skein which could survive the backwashing process, as he described it.

Amended claim 13 now specifies a filtration process requiring the distinctive steps of introducing cleansing gas between the fibers, vertically scrubbing the fibers, keeping them clean, and simultaneously (in the final step) withdrawing permeate.

The rejection in paragraph 2 in the Detailed Action of the office action (page 3) of claims 1-14 under the judicially created doctrine of obviousness-type double patenting over claims 1-20 of U.S. Patent No. 5,639,373 is now moot in view of the terminal disclaimer filed herewith.

The rejection in paragraph 3 (it is misnumbered 2) in the Detailed Action (page 4) of claims 1-14 under the judicially created doctrine of obviousness-type

double patenting over claims 1-20 of U.S. Patent No. 5,639,373 in view of Cote et al. U.S. Patent No. 5,248,424 is respectfully traversed. The thrust of the '424 invention is to maintain an **arcuate configuration** of the fibers in the skein so that the fibers would intercept the rising of bubbles of scrubbing gas. Furthermore, the '424 reference could not have suggested the critical "slack" required in a **vertical** skein since it is clear from its disclosure that the arcuate configuration could reasonably expect to require fibers which were substantially longer than 5% more than the distance between the spaced-apart headers. Nor could the '424 reference suggest the flexible support means between adjacent fibers. The primary reference is inapplicable in view of the terminal disclaimer. With respect to combining the references for the purpose of an obvious rejection, there must be some motivation in the references to make the combination suggested. Neither reference alludes to the problem solved by the invention claimed. Therefore the combination fails to provide an effective rejection for obviousness.

With respect to paragraph 4 (misnumbered 3) in the Detailed Action, it is readily conceded that passing gas in a vertical hollow fiber arrangement to clean the membrane, as taught in Cote et al (U.S. Patent No. 5,607,593) is conventional; however, the '593 reference does not suggest the use of critically "slack" fibers to avoid breakage.

In U.S. Patent No. 5,480,553, Yamamori et al disclose a prior art cylindrical module (in Fig 16) used in their example 1. How their prior art module was constructed is described as follows: "A cylindrical module of the construction illustrated in Fig 16 was fabricated by fixing the ends of porous polyethylene hollow fibers (commercially available . . . . outer diameter of 380  $\mu\text{m}$ ) with a urethane resin. Each hollow fiber form in a loop had a length of 760 mm and a total effective filtering area of the module was 5  $\text{m}^2$ ." If not self-evident from the Fig 16, this description clearly indicates that there is only one header in which both ends of each fiber are potted in a resin so as to form a loop with each fiber. The construction of the article is unrelated to the claimed vertical skein.

In U.S. Patent No. 5,403,479, Smith et al disclose a frameless arrangement of



hollow fibers in spaced-apart headers but provide no suggestion of the problem solved in the invention claimed herein - which problems are specific to a vertical skein used with an aerator so as to simultaneously filter substrate yet maintain the outer surfaces of the fibers clean.

In view of the terminal disclaimer filed herewith, the foregoing remarks, arguments, and amendments to the specification and the claims, it is respectfully submitted that the basis for the rejection has been overcome and that the claims are in condition for allowance.

Respectfully submitted,



Alfred D. Lobo (Reg. No. 24,109)

CERTIFICATE UNDER 35 U.S.C. 1.8(a)

I certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner of Patents and Trademarks, Washington, D.C. 20231, on this 8th day of January 1998.

Alfred D. Lobo  
Attorney for Applicant(s)  
Registration No. 24,109



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